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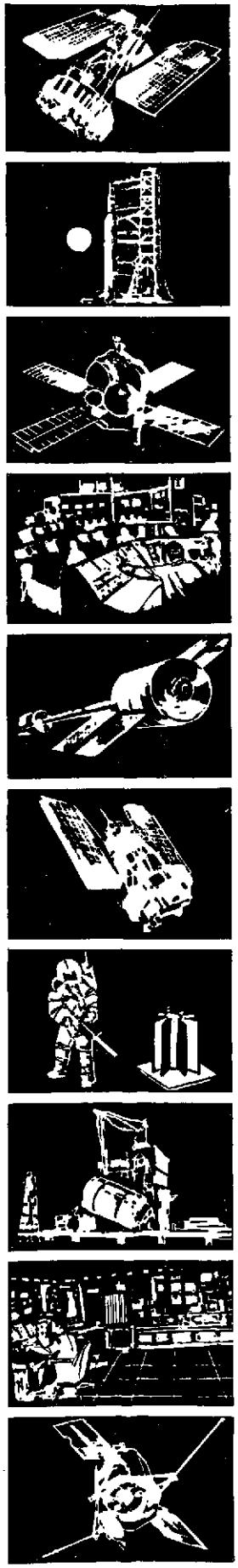

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STUDY FOR IDENTIFICATION OF BENEFICIAL USES OF SPACE

(PHASE II)

254

FINAL REPORT
VOLUME II, BOOK I
TECHNICAL REPORT

CONTRACT NAS8-28179

NOVEMBER 1, 1973

SUBMITTED PER DPD #296, DR #MA-04

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STUDY FOR
IDENTIFICATION OF
BENEFICIAL USES OF SPACE (B.U.S.)
(PHASE II)

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FINAL REPORT - VOLUME II, BOOK I

TECHNICAL REPORT

November 1, 1973

Submitted Per DPD #296, DR #MA-04

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PREFACE

This Final Report on Phase II of the Study for Identification of Beneficial Uses of Space (B.U.S.) is comprised of two volumes:

Volume I - Executive Summary

Volume II - Technical Report

Volume II is further subdivided:

Book 1 - Sections I through IV, Introduction, Method of Study, Study Results, Conclusions and Recommendations

Book 2 - Section V, Appendices A through D

General Electric's Space Division, under contract from the NASA's Marshall Space Flight Center completed Phase I of the Study in December, 1972, and a Final Report for that phase was issued shortly thereafter.

In Phase II, conducted from December, 1972 to December, 1973, the Study has progressed to the investigation of the technology and programmatics involved in development of four of the products selected from those identified by User organizations who participated in Phase I. The selected Products/Organizations were:

- o Surface Acoustic Wave Components.....GE, Electronics Laboratory
- o Transparent Oxides.....Corning Glass Works
- o High Purity Tungsten X-Ray Targets...GE, Medical Systems, Business Division
- o High Specificity Isoenzymes.....Polysciences, Inc.

The methodology employed in this investigation and the results of that effort are reported herein.

The participating organizations supported GE Space Division efforts by evaluating more than 30 major alternative candidate approaches for producing the above products, selecting specific processes for further study, and identifying requirements for nearly 70 experiment and test series necessary to the development of the selected processes.

Subsequent assembly of preliminary program timelines and milestones for such developments revealed a "comfortable" schedule of analyses, experiments and tests, design, development testing and fabrication of operational equipment; with reasonable tolerances in timing to allow for early redesigns, major retests, re-evaluation or results, and moderate redirection; and culminating in 1982-1983 operational dates.

In addition to those Key Individuals from the participating User organizations who contributed specific product, process and planning data in each of their respective areas, the Study Manager acknowledges the considerable contributions of Mr. U. Alvarado and Mr. M. Clarke of the Study Team in analyzing and organizing the wealth of data accumulated; Mr. R. Spencer, the MSFC C.O.R. for the Study, in planning and directing the overall effort; and Mr. G. Wouch and Dr. D. Ulrich, of GE's Space Sciences Laboratory, in providing supporting space processing data.

As noted in the Phase I Final Report, publication of this Phase II report neither implies NASA endorsement of any specific product, process or experiment identified during this phase of the Study, nor a NASA commitment to pursue any program defined as part of this Study.

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SECTION I

INTRODUCTION TO THE TECHNICAL REPORT

This volume of the Final Report on Phase II of the Study for Identification of Beneficial Uses of Space presents the consolidated information developed during a nearly one-year effort.

Previous interim reports, prepared for Performance Reviews during the course of this Phase II effort, have documented preliminary results, conclusions and recommendations derived from the on-going studies. This portion of the Final Report is a recapitulation of that information, updated with the latest data issuing from the final stages of the Study, and backed by appropriate rationale and analyses. Key analyses have been provided in the Appendices collected in Book 2 of this volume.

The purpose of the Technical Report is to provide, for NASA and for interested potential Users, the following:

1. Discussion of methodology for selecting from among potential candidate space processing approaches, definition of requirements for experiments and tests needed to acquire sufficient knowledge to enable proof testing of selected processes, formulation of research and development schedules to achieve proof testing, and documentation of the decision processes involved in such programs;

2. Exposition of the results of applying the above methodology to four specific products identified in the Phase I Study as potentially achieving significant improvements through space processing; and

3. Documentation of additional information (beyond that recorded as a result of the Phase I Study) to the baseline for planning space processing applications

experiments, establishing space processing applications program plans, and initiating space processing business plans.

The currentness of data, analyses, etc. incorporated in this Report requires that it supersede all previously issued reports, presentations, etc.

I.1 REVIEW OF STUDY BACKGROUND

The Phase II Study, reported herein, is a direct outgrowth of the Phase I Study[1] completed in December, 1972. In that early phase of the Study, the scope of effort was broad, in keeping with the Phase I Theme (Figure I-1) and Objectives (Figure I-2).

IDENTIFY PRODUCTS, PROCESSES OR SERVICES THAT WILL BE BEST DEVELOPED OR PRODUCED IN THE UNIQUE ENVIRONMENT OFFERED BY FUTURE SPACECRAFT

AND WILL BE USED DIRECTLY ON EARTH

Figure I-1. Phase I Study Theme

(1) _____Study for Identification of Beneficial Uses of Space (Phase I), Final Report, GE Document #73SD4259, December 10, 1972 and April 23, 1973.

- IDENTIFY SPECIFIC USERS OF SPACE KNOWLEDGE AND CAPABILITIES
- IDENTIFY SPECIFIC KNOWLEDGE OR CAPABILITIES REQUIRED BY USERS
- CORRELATE KNOWLED OR CAPABILITIES WITH USERS
- ASSESS POTENTIAL BENEFITS TO BE DERIVED FROM USING KNOWLEDGE AND CAPABILITIES

Figure I-2. Phase I Objectives

The search to "Identify" "Specific" products, Users, required knowledge, etc. covered many areas of the country, some 80 organizations in 14 of the Nation's 27 basic industries, and dialogs with over 400 key individuals.

That effort was successful, and from over 100 potential ideas offered by such individuals, 12 Ideas appeared to possess considerable merit. The Final Report on Phase I documents rationale, benefits and brief resumes of timing associated with these Ideas.

At the conclusion of Phase I, the NASA Advisory Group for the Study recommended, among other things (Figure I-3), continued activities to assure that Ideas for potentially beneficial space processes be furthered, and that momentum gained during the Study, in developing the interest and participation of a group of non-aerospace Users not be allowed to dissipate. Such an intent, it was felt, might well be served by probing deeper into several of the Ideas developed in Phase I.

- "DON'T STIR THE POT AND NOT FOLLOW UP."
  - MAINTAIN AND EXTEND USER CONTACTS
  - ANALYZE NEXT LEVEL OF STUDY IDEAS

- REVIEW RELEVANT NASA/GOVERNMENT POLICIES/POSTURE

- PUBLICIZE POTENTIAL AVAILABLE KNOWLEDGE/CAPABILITIES

VS

POTENTIAL PRODUCTS, PROCESSES, SERVICES AND BENEFITS

Figure I-3. Advisory Group Recommendations

I.1.1 STUDY RATIONALE

In accordance with the Advisory Group's suggestions and based on recommendations offered in the Phase I Final Report, as approved by the C.O.R., the Phase II Study was planned to meet the intent as expressed in the rationale pictured in Figure I-4.

The emphasis on "Specifics" and the role of "Users" continued to be a major groundrule in Phase II. The success of that mode of operation, which proved successful in Phase I, was further substantiated in Phase II, where the definitive implementation approaches, the evaluation of those approaches, much of the state-of-the-art analysis and experiment requirements, as well as the timelines and decisions could not have been accomplished without the direct involvement of the Users.

1. PREPARATION FOR EVENTUAL IMPLEMENTATION OF SELECTED IDEAS BY:
   - EMPHASIS ON SPECIFIC IDEAS
   - DEFINITION OF SPECIFIC REQUIREMENTS

AND

2. MAINTENANCE OF NON-AEROSPACE USERS INTEREST AND ENCOURAGEMENT OF THEIR PARTICIPATION IN NASA APPLICATIONS PROGRAM BY:
   - EMPHASIS ON SPECIFIC USERS
   - USER AID IN FURTHER DEFINITION.

TO RESULT IN

A. TECHNICAL DATA:
   - APPROACHES FOR IMPLEMENTATING SELECTED IDEAS
   - REQUIREMENTS FOR EXPERIMENTS TO VERIFY APPROACHES
   - MISSION PROFILES FOR SUCH EXPERIMENTS

AND

B. PLANNING DATA:
   - TIMING AND EVENTS FOR R&D THROUGH OPERATIONAL IMPLEMENTATION
   - KEY MANAGEMENT CONSIDERATION RELATED TO SUCH TIMING AND EVENTS.

Figure I-4. Phase II Study Rationale

That emphasis, aimed at producing realistic technical and planning data, enabled the accomplishment of the pertinent Advisory Group recommendations. It continued to foster the Aerospace/Non-Aerospace communities dialogs, and it focussed the Study Team efforts on developing additional data in areas that will be crucial to the practical transformation of what were simply Ideas in Phase I to what will very likely be commercial space-processed products in the 1980's.

I.1.1.1 Study Objectives

The rationale discussed above was supplemented by a set of Objectives set forth in the scope of work of the Statement of Work for the Phase II Study. Those Objectives are given in Figure I-5.

FOR FOUR SELECTED IDEAS FROM PHASE I:

- SELECTION OF BEST APPROACH FOR IMPLEMENTATION OF EACH IDEA
- DEFINITION OF REQUIREMENTS FOR EXPERIMENTS TO VERIFY SELECTED APPROACH - INCLUDING MISSION PROFILES, TYPES OF VEHICLES AND GROUND FACILITIES
- ESTABLISHMENT OF TECHNICAL TIMELINES AND MILESTONES TO ACHIEVE OPERATION (PRODUCTION, OR SERVICE) OF PROTOTYPE FACILITY/PILOT PLANT
- FORMULATION OF PLANNING PROFILES TO RELATE KEY MANAGEMENT DATA TO TECHNICAL TIMING. DATA TO INCLUDE DEVELOPMENT STEPS, DECISION POINTS, ALTERNATIVES, RISKS, MAJOR FACILITIES, UNIQUE MANPOWER

Figure I-5. Objectives - Phase II

Starting with the relatively broad information available from Phase I for the four Ideas approved for Phase II by the C.O.R., the Study aimed at satisfying these objectives:

I.1.1.1.1 Selection of Best Approach

Our goal was to provide data and the logic for processes or steps in processes which would be performed in implementing the four Ideas and which would require performance in the orbital environment, or would be beneficially affected by such performance. Consideration was to be given to both ground- and space-based alternatives for each process and each step in each process. The "best approach" was to be developed by evaluation against significant scientific, engineering and business criteria. Over 130 major and minor alternatives for the processing of the four selected products were defined, and most of these were readily resolved. However, the evaluation against these criteria was carried out on, primarily, a judgemental basis, and the lack of available information for many areas of new technology coupled with varying opinions on technology growth, would not allow the clear resolution of some 10% of the alternatives. Such unresolved issues subsequently became the subjects of identified experiments and tests required to substantiate the selected approaches. Typically, selections could not be justified for ground-based versus orbit-based preparations of gels for isoenzymes separation, ground-based versus orbit-based shaping of the tungsten material, heating rate for the transparent oxides, etc.

I.1.1.1.2 Definition of Requirements for Experiments

This objective directed examination of processes and process steps of the selected approaches against present state-of-the-art, and the definition of requirements for experiments/tests to provide data necessary to the orderly development of the selected approaches. Judgement of present state-of-the-art in many phenomenological and process areas indicated that "gaps" in available data would require filling before enabling Users to proceed with implementation of most approaches. More than 40 of such state-of-the-art "gaps" were covered in the dialogs between

the Study Team and the four User organizations. Such gaps included heating and convection in large pore gels during long duration electrophoresis at voltage gradient < 10 volts/cm, effects of containerless supercooling on tungsten, etc. The Space Division Study Team learned much about the technology involved in the four product areas of the Study during this stage of the Study, and the Users accrued considerable knowledge of aerospace facilities, testing techniques and planning methods. Such cross-fertilization may well be one of the most important aspects of this type of interaction.

I.1.1.3 Establishment of Timelines and Milestones

This objective was aimed at providing the preliminary supporting technical planning data base for programs to implement the selected approaches for the four products under study. A 1982 shuttle flight was given as a baseline for the production phase of the selected products. The initial scheduling of the research and development effort for all four products, which must precede that phase, indicates that such a date allows sufficient time for an orderly sequencing of key program elements without the need for parallel, time saving but costly, efforts. Furthermore, there appears to be sufficient time and flexibility to acquire, analyze, and absorb data from earlier experiments or analyses before initiating a subsequent effort, and to redirect efforts or retest a thesis in the event that unexpected results are obtained.

I.1.1.1.4 Formulation of Planning Profiles

It was recognized, prior to initiating this Phase of Study, that implementing the programs which meet the preceding objective would require many administrative decisions prior to, and during, the programs. This objective was aimed at defining the content and flow of such decisions as well as their interrelationships and relationships with technical decisions. To assure the reality of the Study

results in support of this objective, the participating Users imposed their own business planning groundrules in formulating such decisions, estimating the risks involved in each decision, and indicating their preferred alternatives.

I.2 THE FOUR PHASE I IDEAS STUDIED IN PHASE II

To develop the comprehensive data called for in the rationale and objectives governing the Phase II Study, it was necessary to limit the number of Ideas to be studied. An analysis of the twelve high merit Ideas of Phase I was carried on by the Phase II Study Team to ascertain which Ideas would best serve the aims of the NASA Applications Program, reflect the broad community of potential space processing Users, offer meaningful potential benefits as a result of their implementation, and respond to the Advisory Group recommendations. With the aid of the NASA C.O.R. for the Study, four Ideas were identified for further study.

- o Separation of Isoenzymes
- o Processing of Tungsten X-Ray Targets
- o Processing of Transparent Oxides (Formerly Amorphous Oxides)
- o Fabrication of Surface Acoustic Wave Components

Each of these Ideas is disucssed in considerable detail in the Phase I Final Report. A short summary of each is given below.

I.2.1 SEPARATION OF ISOENZYMES (Figure I-6)

Separation of Isoenzymes is an Idea for utilizing the "zero G" of orbital flight to eliminate convective forces and other gravity-induced effects from a process employed to perform separations of biological materials. The process is, thereby, expected to result in more specific separations, thus, purer preparations of the isoenzymes.

POTENTIAL APPLICATIONS - SPECIFIC ISOENZYMES (RNA'S, DNA'S OR OTHER MOLECULES WHICH ARE PRESENT IN DISEASE STATES) TO BE USED FOR PRODUCTION OF SPECIFIC ANTIBODIES WHICH AID IN DIAGNOSIS (AND TREATMENT) OF DISEASES

USERS - PHARMACEUTICAL COMPANIES, HOSPITALS, CLINICS, PHYSICIANS

KEY INDIVIDUALS - DR. B. HALPERN, POLYSCIENCES INC.

USER GOAL - MANUFACTURE OF PURE ISOENZYMES ON A PREPARATIVE (SEVERAL HUNDRED MILLIGRAM) SCALE

PROBLEM AREAS - SEPARATION OF UNDENATURIZED ISOENZYMES, (STORAGE OF SAMPLES, SURVIVAL OF EQUIPMENT AND GELS OF LAUNCH CONDITIONS)

CAPABILITIES REQUIRED - "GENTLE" ELECTROPHORETIC SEPARATOR, PRESERVATION SYSTEM

APPLICABLE SPACE PROPERTIES - ZERO GRAVITY (REDUCED THERMAL CONVECTION, SEDIMENTATION)

SPECIAL REQUIREMENTS - IF NECESSARY, PREPARATION OF GELS, LOADING OF SAMPLES, AND ISOLATION OF PRODUCTS IN ORBIT

Figure I-6. Separation of Isoenzymes
(And Other High Molecular Weight Biologicals)

Such pure enzymes are expected to carry on, and extend the number of, the several types of diagnostic procedures developed over the past few years for such difficult-to-diagnose-in-early-stages problems as cardiac infarction, hepatoma (and other cancers), sickle-cell anemia, etc.

I.2.2 PROCESSING OF TUNGSTEN X-RAY TARGETS (Figure I-7)

The production of higher quality tungsten x-ray targets is not only an immediate need, but it also appears to be relatively within the foreseeable state-of-the-art. The User has a continuing material development program in place, into which this Phase of the Study has been fit.

Processing tungsten via containerless melting and supercooling is expected to provide a fine grain structure not possible with the powder metallurgy techniques in use in ground facilities.

POTENTIAL APPLICATION - X-RAY TUBES

USERS - HOSPITALS, CLINICS, AND OTHER HEALTH SERVICE ORGANIZATIONS

KEY INDIVIDUALS - WILLIAM D. LOVE - GE MEDICAL SYSTEMS DIVISION

USER GOAL - MANUFACTURE TUNGSTEN TARGETS PROVIDING X-RAY TUBES WITH LONGER LIFE AND LESS PERFORMANCE DEGRADATION WITH TIME

PROBLEM AREA - TARGETS DEVELOP SURFACE FRACTURES WHICH TRAP X-RAYS AND INITIATE TARGET FAILURE

CAPABILITIES REQUIRED - MELT AND SOLIDIFICATION PROCESS WHICH REDUCES IMPURITIES WHICH CAUSE LOW DUCTILITY. IMPROVEMENT OF GRAIN STRUCTURE OF TUNGSTEN TARGET

APPLICABLE SPACE PROPERTY - ZERO GRAVITY LEVITATION MELTING TO ELIMINATE CONTAMINATION AND NUCLEATION FROM CRUCIBLE

Figure I-7. Tungsten X-Ray Targets

Potential economic and social benefits of the successful development of structurally better tungsten for x-ray targets have made this a key study idea.

I.2.3 PROCESSING OF TRANSPARENT OXIDES (Figure I-8)

Originally, this Idea was called Amorphous Oxides. The new title does not imply a new effort, merely that even transparent polycrystalline forms of the subject oxides would be valuable. The advantages of new forms of oxides produced in the space environment through containerless melting and supercooling to avoid devitrification are likely to be in the optical and strength properties.

This Idea is highly speculative. Only the most meager data is available to indicate the possibility of success. The experimental program defined in subsequent sections of this Report is aimed at providing more definitive data as to such possibilities. In any case, the Aerospace/Non-Aerospace dialogs involved in investigating this Idea should prove mutually beneficial to NASA and the User.

POTENTIAL APPLICATIONS	INDUSTRIAL AND SCIENTIFIC WINDOWS, LENSES ULTRAVIOLET, VISIBLE & INFRARED
USERS	OPTICAL INSTRUMENT AND LIGHTING EQUIPMENT MANUFACTURERS
KEY INDIVIDUALS	GEORGE MC LELLAN, CORNING GLASS WORKS
USER GOALS	PREPARATION OF LENSES, WINDOWS OF TRANS-PARENT OXIDE MATERIALS AND SPINELS HAVING STRENGTH AND TRANSMISSIVITY PROPERTIES SUPERIOR TO SIMILAR MATERIALS NOW BEING PRODUCED ON EARTH.
PROBLEM AREAS	SOLIDIFICATION WITHOUT DEVITRIFICATION, HIGH PURITY
CAPABILITIES REQUIRED	NO CONTAMINATION OF MELT, NO SOLID/LIQUID INTERFACE DURING SOLIDIFICATION
APPLICABLE SPACE PROPERTIES	OXYGEN AND WATER-FREE ATMOSPHERE, ZERO GRAVITY (FREEDOM FROM CONTAINER CONTAM-INATION AND NUCLEATION)

Figure I-8. Transparent Oxide Materials

I.2.4 FABRICATION OF SURFACE ACOUSTIC WAVE COMPONENTS (Figure I-9)

The production of Surface Acoustic Wave electronic components involves both ground and space processes, where space processing provides freedom from gravity effects on crystal growth, the freedom from low frequency vibrations such as those of seismic origin.

POTENTIAL APPLICATIONS	SIGNAL PROCESSORS (WIDEBAND RADAR), DIGITAL ENCODERS (COMMUNICATIONS), BANDPASS FILTERS (RADAR AND COM-MUNICATIONS) ETC., FOR EXTREMELY HIGH FREQUENCIES
USERS	MANUFACTURERS OF RADAR, COMMUNICATIONS EQUIPMENT, AIRPORTS, AIRCRAFT, COMMUNICATIONS COMMON CARRIERS
KEY INDIVIDUALS	DR. S. TEHON, DR. S. WANUGA, ELECTRONICS LABORATORY, GE
USER GOALS	PRODUCTION OF ELECTRONIC COMPONENTS FOR OPERATION AT >30 GHZ
PROBLEM AREAS	OBTAINING LARGE PIEZO AND NON-PIEZO CRYSTALS, CIRCUITRY IMPERFECTIONS CAUSED BY VIBRATIONS IN IMPRINTING SYSTEM
CAPABILITIES REQUIRED	ZERO LOADING AND CONVECTION DURING CRYSTAL GROWTH, ISOLATION FROM VIBRATION DURING IMPRINTING PROCESS
APPLICABLE SPACE PROPERTIES	ZERO "G" AND ISOLATION FROM TERRESTRIAL ENVIRONMENT

Figure I-9. Surface Acoustic Wave Electronic Components

The technical advantages and high economic value of such components, which could operate at frequencies of 10 to 30 GHz appear extremely attractive for radar, communications and other applications.

I.3 SUMMARY OF THE STUDY

The Study has been successfully completed. Results meet all the Objectives, comply with the rationale, and were developed within the Assumptions and Guidelines, documented in the Statement of Work for the Phase II Study.

I.3.1 TECHNICAL ANALYSIS OF SELECTED IDEAS

The definitions of logical alternative approaches for implementing the four product Ideas of this Study form the data base for the remainder of the Study. Dialogs held between the space-oriented Study Team and the product-oriented Users engendered a synergism that produced nearly 30 major alternatives for processing the four products, and more than 100 alternatives for minor areas. Figure I-10 briefly summarizes those alternatives. In all cases, both ground- and space-based process steps were considered, where judged to be of legitimate possibility.

The next Study effort was programmed to reduce the number of alternatives to one approach for each product. That approach was selected on the basis that it was judged the "best" in comparison to other alternatives. For most alternatives, judgement was based on consideration of the technical and business criteria listed in Figure I-11. Not all of these criteria were applicable to all alternatives nor were all criteria of equal value. The dialogs between Study Team and Users first established the pertinent criteria and their relative importance for each alternative and then proceeded to weigh the alternatives against the criteria. Where available data from past experience, laboratory investigations, or space

STUDY ITEM	MAJOR ALTERNATIVE APPROACHES EVALUATED	OTHER ALTERNATIVES EVALUATED (NUMBER, TYPICAL EXAMPLES)
SEPARATION OF ISOENZYMES	SEPARATION VIA: • FREE ELECTROPHORESIS • SMALL PORE GEL ELECTROPHORESIS • LARGE PORE GEL ELECTROPHORESIS • ISOELECTRIC FOCUSSING • CHROMATOGRAPHY • OTHER SMALL FORCES	41 ALTERNATIVES INCLUDING: • PRESERVATION VIA: - FROZEN SOLUTION - LYOPHILIZATION - ADDITION OF ANTISEPTICS • PREPARATION OF GELS: - ON GROUND - IN ORBIT • LOADING OF SEPARATOR: - ON GROUND - IN ORBIT
TRANSPARENT OXIDE PROCESSING	HEATING AND MELTING VIA: • INDUCTION • LASER • ELECTRON BEAM • RF • SOLAR CONCENTRATOR	31 ALTERNATIVES INCLUDING: • LOADING OF MELTING SYSTEM: - ON GROUND - IN ORBIT - AUTOMATIC - MANUAL • POSITION CONTROL VIA: - ELECTROMAGNETICS - ACOUSTICS - RF - GAS JETS - ELECTROSTATICS
TUNGSTEN X-RAY TARGET PROCESSING	LEVITATION MELTING VERSUS FLOAT ZONE REFINING HEATING AND MELTING VIA: • RF • SOLAR CONCENTRATOR • ELECTRON BEAM	27 ALTERNATIVES INCLUDING: • HEATING PROFILE: - SLOW RISE - FAST RISE - DWELL BELOW MELT TEMPERATURE - NO DWELL BELOW MELT - SHORT DWELL DURING MELT - PROLONGED DWELL DURING MELT - NO DWELL DURING MELT • SHAPING VIA: - SPINNING DURING SOLIDIFICATION - GROUND BASED FORMING - MACHINING
FABRICATION OF SURFACE ACOUSTIC WAVE COMPONENTS	IMPRINTING OF CIRCUITRY VIA: • PROGRAMMED ELECTRON BEAM • X-RAY LITHOGRAPHY • PHOTOLITHOGRAPHY • LIFT-OFF PROCESSING CRYSTAL GROWTH VIA: • FLOAT ZONE • CZOCHRALSKI • FLUX • HIGH PRESSURE HYDROTHERMAL • VERNUIL • GROUND VERSUS IN ORBIT	12 ALTERNATIVES INCLUDING: • ULTRACLEANING VIA: - GROUND VERSUS IN ORBIT - BACK SPUTTERING - ION BEAM SCRUB • METALLIZATION VIA: - GROUND VERSUS IN ORBIT - SPUTTERING - VAPOR DEPOSITION • CRYSTAL ACOUSTIC SURFACE: - AS GROWN - CUT AND POLISHED

Figure I-10. Summary of Alternative Processing Approaches

EASE OF PROCESSING

ALLOWABLE TOLERANCES IN ENVIRONMENT CONDITIONS
REQUIRED PRECISION OF PERFORMANCE
DURATION OF PERFORMANCE
COMPLEXITY OF FUNCTIONS

EQUIPMENT REQUIREMENTS

DEGREE OF AUTOMATION FEASIBLE
RELATIVE SIZE, WEIGHT

SUPPORT UTILITIES, SERVICES

VARIETY OF TYPES
RELATIVE AMOUNT

MATERIALS

RELATIVE AMOUNTS OF RAW MATERIALS
RELATIVE AMOUNTS OF WASTES

TIMELINESS

RELATIONSHIP OF AVAILABILITY TO NEED

FINAL PRODUCT

QUALITY
RELATIVE COSTS

VERSATILITY

POTENTIAL FOR OTHER APPLICATIONS

Figure I-11. Criteria for Selection Among Alternative Approaches

experiments provided any solid indication of the benefits (or lack thereof) for the technology involved in a particular process step, judgements were relatively straightforward. Where no data was available, but projections of technology could be agreed upon, judgements were also relatively straightforward, albeit somewhat "shaky". Some areas in the alternatives, however, either revealed conflicting data, or had no basis for judgement or agreement on projected technology. Such process steps, it was assumed, would require experiments and tests for their resolution. A summary of the selections made in this effort, and the specific process steps for which selections were either not well supported or not feasible, is given in Figure I-12.

IDEA	PROCESS STEPS BEST PROGRAMMED IN SPACE	PROCESS STEPS TO BE RESOLVED BY EXPERIMENT/TEST	ALTERNATIVES IN PROCESS STEPS TO BE RESOLVED BY EXPERIMENT/TEST	PROCESS STEPS PREFERRED TO BE PERFORMED IN SPACE
SEPARATION OF ISOENZYMES	SEPARATION	PREPARATION OF GELS, SAMPLES (INCLUDING LOADING OF SEPARATOR).	PROTEIN DETERMINATION VS. ACTIVITY DETERMINATION PACK ISOENZYMES VS. PRODUCE AND PACK ANTIBODIES	- - - -
TRANSPARENT OXIDE PROCESSING	POSITION CONTROL, HEATING (DEGASSING AND MELTING), COOLING, COLLECTION	LOAD LEVITATION SYSTEM	HIGH RATE VS. MODERATE RATE HEATING. MELT TEMP. VS. SUPER HEAT	- - - - -
HIGH PURITY TUNGSTEN X-RAY TARGET PROCESSING	PROCESS CONDITIONS, LEVITATION (POSITION CONTROL), HEAT (OUTGASSING AND MELTING, COOLING	SHAPE THE TARGET	HIGH PURITY VS. COMMERCIAL TUNGSTEN, 0.91 VS. 096 KG TARGET	- - - - -
FABRICATION OF SURFACE ACOUSTIC WAVE COMPONENTS	CRYSTAL GROWING, FABRICATE MASK, DEPOSIT PIEZO FILM	-----	-----	ULTRA-CLEANING METALLIZATION RESIST-COATING MASK ALIGNMENT X-RAY EXPOSURE

Figure I-12. Summary of Selected Approaches

Technical analysis for this Study also included definition of requirements for experiments, and tests which would support programs of development for implementation of the selected approaches. For such definition, it was necessary to review each process step in the selected approaches, ascertain the critical elements in each such process step, match these elements against the state-of-the-art in their related disciplines and technologies, and project any "gaps" in knowledge that could inhibit the development of each selected implementation approach. These gaps, then, indicated the need for appropriate experiments and tests to provide data in support of such development. Over 90 "gaps" were identified, calling for some 66 types of experiments and tests in facilities that ranged from ground laboratories to sounding rockets to the space shuttle. Figure I-13 presents some examples extracted from the results of this effort. It was interesting to note, during this task, that commercial companies frequently eschew sophisticated analytical/technical studies to obtain new state-of-the-art information, in favor of experiments which provide the answers they seek in a limited range of interest. In any case, this method of operation is generally

KNOWLEDGE GAPS	EXPERIMENTS AND VERIFICATION TESTS	EXPERIMENT AND TEST REQUIREMENTS (SUMMARY)
DEGASSING RATES EFFECTS OF TEMPERATURE, VACUUM, INITIAL TUNGSTEN PURITY.	DEGASSING TIMES VS TEMPERATURE, TYPE AND QUANTITY OF GASES, DEGASSING EFFECTS ON MECHANICAL PROPERTIES, INTERSTITIALS CONTENT.	STANDARD GROUND LAB, VACUUM INDUCTION FURNANCE, RF GENERATOR, MASS SPECTROMETER, GAS SAMPLING SYSTEM, PROCESS INSTRUMENTATION, MATERIALS AND GAS ANALYSIS EQUIPMENT, SPECTROSCOPE, 2-1/2 WEEKS PER SPECIMEN, MANUAL CONTROL.
POSITIONING, HEATING, DEGASSING, MELTING, SUPERCOOLING ADVANTAGES, SELECTION OF PROCESS ATMOSPHERE, ASSESSMENT OF PROCESSED TUNGSTEN METALLURGICAL AND OPERATIONAL PROPERTY GAINS.	LEVITATION, HEATING, MELTING TEST FOR R/F AND/OR ELECTRON BEAM AS CONTROL AND HEATER. ALSO EFFECTS OF VACUUM AND INERT GAS ON TUNGSTEN EVAPORATION. ALSO SUPERCOOLING EXPERIMENT.	STANDARD GROUND LAB, EQUIPMENT AS ABOVE PLUS INERT GAS SYSTEM AND COPPER OR CERAMIC COOLING CRUCIBLE. ALSO OPERATIONAL TESTING (TEST X-RAY TUBES, PROGRAMMED TEST APPARATUS, STANDARD SHIELDED TEST CHAMBER, ETC.). 4-1/2 TO 5 HOURS PER RUN PLUS 1 WEEK CHARACTERIZATION PLUS 15 DAYS OPERATIONAL TESTING. MANUAL PLUS AUTOMATED OPERATIONAL TESTING.
ASSESSMENT OF POSITIONING SYSTEM AND ACQUISITION OF DESIGN DATA.	ZERO "G" VERIFICATION TEST OF POSITIONING SYSTEM.	DROP TOWER, KC135, OR SOUNDING ROCKET. TEST PACKAGE OF POSITIONING DEVICE, TUNGSTEN SPECIMEN, CAGING SYSTEM, POSITION MONITORING INSTRUMENTATION, RECORDER, POWER SUPPLY AND DISTRIBUTION, PHOTOGRAPHIC APPARATUS, MEASUREMENT GRID. AUTOMATED. MINIMUM TEST TIME 4 TO 10 SEC - DROP TOWER, 20 TO 40 SEC - KC135, 4 TO 10 MIN - SOUND ROCKET. TELEMETRY OR RECORDING OF DATA, RECOVERY OF TEST PACKAGE AND RECORDER DATA.
FINAL PROCESS DESIGN DATA DEFINITIONS.	ZERO "G" PROCESS VERIFICATION TEST.	SHUTTLE FACILITY. TEST PACKAGE AS ABOVE, AUGMENTED FOR MULTIPLE SAMPLES, EXTENDED TEST TIMES, AND COMPLETE PROCESS INSTRUMENTATION. PROVISION FOR CREW OBSERVATION, MONITORING AND PROCESS MODIFICATION. AUTOMATED PROCESS, MANUAL RECYCLE. 1-1/2 - 3 HOURS PER RUN, PLUS 15 DAYS OPERATIONAL TESTING. TELEMETRY OF DATA, RECOVERY OF TEST PACKAGE AND PROCESSED SAMPLES.

Figure I-13. Typical Requirements for Experiments to Verify Selected Approach for High Purity Tungsten X-Ray Targets

a function of the time and skills available, as well as the costs involved. In the course of defining experiment requirements, the Study Team introduced the concept of "low cost testing" to the four non-aerospace Users. Briefly, the thesis of low cost testing is that certain experiment and test requirements could likely be satisfied in test facilities more or less unique to the aerospace industry and relatively inexpensive to operate, compared to the past space experiments with which the public (and the Users) have some slight acquaintance. On that basis, the Users were requested to review their experiment/test requirements against the characteristics/capabilities of a set of potential test facilities, listed in Figure I-14, and to establish, where possible, their requirements for experiments and tests such that significant portions of those requirements could be met in low cost facilities. To aid in that effort, summary documents and dialogs concerning the physical, environmental, and financial aspects of these facilities were provided. While emphasis was placed on drop towers, zero "G"

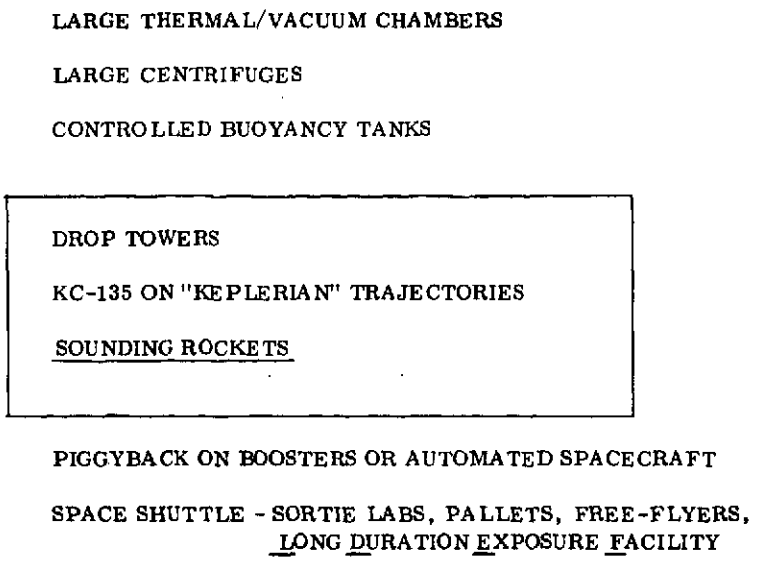

Figure I-14. Low Cost Facilities for Obtaining or Simulating Space "Properties" and Space Flight Conditions

aircraft trajectories, and (mainly) sounding rockets, this familiarization effort did not neglect facilities at either end of the spectrum - ground and space. As a result of this effort, more than 2/3 of the defined experiments/tests appear to be compatible with facilities that range from gound laboratories to sounding rockets.

The final step in technical analysis has been the construction of mission profiles for the performance of the identified experiments and tests. Since only generic experiment/test facilities were identified in the Study, these mission profiles were relatively gross, and were concentrated on the experiment/test operations portions of these missions.

I.3.2 TECHNICAL PLANNING DATA FOR IMPLEMENTING SELECTED APPROACHES

Based on the selection of an approach for each of the four Ideas under study, and on the definition of a set of experiments and tests required in order to implement each approach, the next task was to formulate a logical schedule of the full scope of technical and administrative efforts necessary to carry each

Idea from the present conceptual stage to the point of eventual production in the 1982 time frame. The "know how" of the Users in the specific product technologies, business and organization methods was coupled, through dialogs, with the Study Team experience in space processing technology, space program planning methods, etc. to provide such schedules. The timelines and program milestones formulated in that effort, show a not-surprising considerable diversity among the four products. On the other hand, as pictured in Figure I-15, there are some similarities. Analyses, for example, where felt to be of value, have already started; Drop Tower tests are concentrated mainly in 1975; all the approaches expect to be ready for verification tests on early shuttle flights in 1979-1980; initial organizational activities would be carried on in the coming year; initial NASA/User financial arrangements should be developed in the next six months.

- ANALYSES STARTING IN 1973
- LABORATORY TESTING LATE 1973, HEAVY IN 1974 - 1976, SOME TO 1979
- DROP TOWER TESTS 1974, MAINLY 1975
- KC-135 TESTS 1975, MAINLY 1976, SOME IN 1977 - 1978
- SOUNDING ROCKET TESTS, SOME IN 1975 - 1976, HEAVY IN 1977, SOME IN 1978 - 1979
- PRESHUTTLE SPACECRAFT TESTS, 1975, 1977, HEAVY IN 1978 - 1979
- SPACE SHUTTLE TESTS, HEAVY IN 1979 - 1980, SOME THROUGH 1983
- SUPPORT EQUIPMENT AVAILABILITY, MID-1978 TO MID-1983
- PROTOTYPE/PILOT PLANT DEMONSTRATIONS, MID-1981 TO MID-1982
- GROUND FACILITY PREPARATION, SOME 1975 TO 1977, 1979 TO 1982
- FULL-SCALE OPERATIONS BEGIN, 1982 TO MID-1983
- ADMINISTRATIVE AND TECHNICAL ORGANIZATION SET UP, 1973 - 1974, LATER MODIFICATION
- FINANCIAL ARRANGEMENTS DEFINED: INITIALLY, LATE 1973, EARLY 1974; FINAL, 1975, 1978 - 1980

Figure I-15. Summary of Development Program Highlights

I.3.3 MANAGEMENT PLANNING DATA FOR IMPLEMENTING PROGRAMS

The previously formulated timelines and milestones only lightly touched upon the non-technical aspects of implementing the selected approaches for producing the four products under study. NASA, however, recognizes that decisions to go

ahead with commercial ventures in space will involve a considerable number of management decisions, interlaced in timing with technical decisions. It was, therefore, necessary to develop the overall flow of decisions involved. Once again, dialogs between the Study Team and Users, each representing their own ranges of expertise, enabled the assembly of consistent decision flows, identification of the major alternatives involved in the decisions, estimation of the probabilities that such alternatives would occur, and the Users indication of the alternatives he favored. The decision flows, reflecting the sequence of decisions as well as prerequisite decisions (where applicable) provide a rapid display of "nodes" (or critical decisions) in the program, and thus establish areas of emphasis for both NASA and the User. The alternatives and associated probabilities, which are frequently very much a matter of opinion, and which can, in some cases, be restructured in various permutations and combinations, could serve as the focal point for NASA/User negotiations, if further steps in the implementation programs are contemplated. The Users' initial positions in such negotiations are indicated for each listed decision.

I.4 SIGNIFICANT POINTS IN THE TECHNICAL REPORT

This volume of the Phase II Final Report documents a number of key points which support the general conclusions listed below. These key points are noted and referenced according to the specific Figures and Sections which discuss them in more detail.

1. Continuation of the Phase I Relationships (Dialogs, Mutually Supportive Analyses) maintained the Users' interest and encouraged their participation in NASA's Applications Program. The more than 130 alternative approaches generated

by the participating Users, as illustrated by the discussions in Sections III.1, III.2, III.3 and III.4, and summarized in Figure I-10, is ample evidence of that interest and participation.

As a further example of that interest, General Electric's Medical Systems Business Division has committed, in writing, the equivalent of about $30,000 of Company funds for raw materials, test x-ray tubes, manpower, etc. to perform the initial operational testing of improved tungsten materials formed by initial experiments of levitation melting, in the event NASA initiates such an experiment program.

2. The Phase II Study has furthered the understanding and information that would be required to prepare for eventual implementation of the four Ideas under study. Sections III.5, III.6, III.7 and III.8 provide candid judgements of the lack of hard data on various process steps in the selected approaches. The definition of requirements for nearly 70 series of experiments, given in these sections, documents details, not heretofore available, of additional required effort for implementation of the Ideas under study. In addition, the details presented in the timelines and milestones of Sections III.10.1, III.10.2, III.10.3, and III.10.4 enable, for the first time, both NASA and the User to establish initial plans for integration with other programs in their respective futures. Finally, the decision flows in Sections III.11.1, III.11.2, III.11.3 and III.11.4 pinpoint, for the first time, the timing, content, importance, and probabilities involved in decisions to pursue implementation of the Ideas under study.

3. In general, all the technical and most planning aspects of "relating the User's versions of product-.....oriented implementation, experimentation, production or operation, and planning to the limitations, capabilities, timing,

alternatives and planning which must be accounted for in specific space programs" (Quote is from Section IV, Expected Results, in Statement of Work for Phase II) showed many "points of agreement, compromise" and no "incompatibility". The accommodation of experiment requirements via available potential test facilities discussed in Sections III.5, III.6, III.7 and III.8 reflects that agreement and compatibility. Further examples are visible in the timelines and milestones shown in Sections III.10.1, III.10.2, III.10.3 and III.10.4. The only potential incompatibilities, which would be very readily resolvable by compromises in timing, are those experiments and tests calling for flights in automated spacecraft prior to shuttle operations. If "piggyback" space were not available for such experiments and tests, they could be delayed until shuttle accommodations were available, and the resulting schedule slippage would not be catastrophic.

4. On the other hand, some of the planning aspects in the results of the Phase II Study surface some potential "points of incompatibility". These appear in the decision flows of Sections III.11.1, III.11.2, III.11.3 and III.11.4, and stem from the timing, the User-preferred alternatives related to financial and legal arrangements. Since it is too early to indicate the specifics of such arrangements, and since negotiations toward compromises on such issues remain for future effort, any incompatibilities that may be indicated must be listed as "potential".

5. Finally, the Phase II Study reaffirms a major conclusion of Phase I, which is reprinted here verbatim:

"Considerable analytical and experimental work must be accomplished before any of the Ideas documented herein can be considered feasible. The Phase I Study called for "Identification of Beneficial Uses of Space" (Figure I.1)[1]. The

(1) _____Study for the Identification of Beneficial Uses of Space (Phase I), Final Report, GE Document #73SD4259, December 10, 1972, and April 23, 1973.

implication, subsequently reaffirmed in NASA Steering Group Reviews, was that in-depth, technical "analysis" of such uses was not part of the Study. The breadth of Users, goals, problem areas, Ideas, etc., to be investigated within the funding and time available precluded all but the most rudimentary investigation into the technology and disciplines involved in the Ideas identified. Although Section V - Appendices[1] provides certain preliminary analyses, the bulk of detailed analyses, particularly in such disciplines as fluid mechanics, thermodynamics, vibration, etc., must issue from later studies. For this Phase of Study, an Idea was accepted for "Identification" if it met all the Study requirements for "specific-ness"; if, in the judgement of the Study Team, the problems, needs, and issues that presently inhibited its completion could be justified technically by the experience of originating Key Individuals and other consultants; and if the Study Team could find a logical, potential match between such problems, needs or issues and the projected capabilities or knowledge our experience indicated could possibly be obtained in space. Thus, in lieu of the surer, more costly, more time-consuming technical analyses, we substituted User experience, and Study Team space judgement. In that light, it behooves the reader to exercise care in the use of the data contained herein."

The experiment requirements referenced in Paragraph 2. above reaffirms the preceding statement, and the analytical efforts shown in the timelines of Sections III.10.1, III.10.2, III.10.3 and III.10.4 as well as the fact that 1/3 of the experiments and tests required are to be performed in ground laboratories indicate the extent of the previously noted "considerable analytical and experimental work.

Conclusions on specific details of this Phase of the Study will be found in Section IV of this report.

I.5 SUMMARY OF RECOMMENDATIONS

In the course of the Phase II Study, discussions with members of the NASA Steering Group for the Study, members of the Materials Advisory Board, the Study C.O.R., other NASA personnel at MSFC and OA, as well as with potential Users, both those involved in the Study and others not so involved, have indicated the possible value of additional effort related to this Study. Such interchanges form the basis of the recommendations summarized below. We recommend that NASA proceed with:

1. Performance of a Business Planning Study. Further encouragement of commercial utilization of space processing, and furthering of NASA/User relationships, would accrue from a Business Planning Study of the four products studied in Phase II. The aim of such a study should be to assess the validity of standard business planning and market forecasting methods for determining the cost and resource requirements of space processing these four products.

2. Promotion of Space Processing for Non-Aerospace Users. The establishment of a broadly-based constituency for space processing will gain significantly, based on experience during the Phase I Study, from a low keyed educational campaign among potential User communities. The Phase I effort showed that the "gestation" period for space processing Ideas in the non-aerospace communities is relatively long. Articles on space processing in the non-aerospace technical journals and direct mailings of significant information to specific non-aerospace organizations and key individuals could be a part of such a campaign. Results from experiments on Skylab, from MSFC laboratory tests, and various studies under contract could initiate the desired NASA/User contacts. A major step in furthering such a relationship would be the

initiation of an information exchange mode, in which NASA and potential Users could familiarize each other with space processing capabilities and specific ground process problems, respectively.

3. Identification of Additional Beneficial Uses of Space. In conjunction with 2. above, an extension of the Phase I effort would certainly add to the list of identified specific Users. During the Phase II Study, several previously uncontacted organizations, through information supplied by other sources, established contact with the Study Team, requested information and briefings, and, on two occasions, visited the Space Division, all for the purpose of assessing the possibilities of space processing for their products. Several potentially high merit Ideas subsequently resulted from these contacts. A low level, but consistent, effort in this area should provide further such useful data.

4. Initiation of Experiment Programs. The Phase II effort on the four subject products identified some 22 series of ground laboratory experiments and tests in other low cost facilities, all of which must precede testing in the full space environment. A certain approach to establishing the reality of NASA's applications program for non-aerospace Users would be to initiate the earliest required low cost tests and experiments. Present formalities and documentation, which are required prior to initiating such a test program, should be reviewed to determine whether simplification could not be introduced in order to make participation by non-aerospace Users more attractive.

SECTION II

METHOD OF THE STUDY

As in the Phase I Study, it was anticipated that the essence of the Phase II effort would be the melding of aerospace expertise with the expertise intrinsic in each of the four product areas under study. Furthermore, that melding was to be accomplished through the same media utilized in the Phase I Study - dialogs and mutually supportive analyses between the Study Team and the User organizations. With that background, the prescribed Rationale (Figure I-4) and Objectives (Figure I-5) for the Study, there remain only the Study Logic and a set of specific tasks to define the methodology of the Phase II effort. These are described below.

II.1 STUDY LOGIC

The Study was carried out in the three steps depicted in Figure II-1. After approval of the four Ideas by the C.O.R., the Study Team initiated dialogs with Users in order to commence work on Step I of the Study. As shown in the figure, such dialogs and necessary analyses superimposed the space environment and processes knowledge of the Study Team on the methods, techniques, state-of-art, etc., unique to the Idea, as known to the User. Lessons learned from the earlier Phase I Study and the Study Team/User relationships developed during that Study had shown that the give and take of dialogs was a highly satisfactory method of uncovering the technical compatibilities and incompatibilities between what the User felt was required and what space programs could provide. Utilization of the knowledge gained during the previous phase and updating of that knowledge through analysis of current literature, as in the earlier effort, enabled the Study Team to initiate this step at the outset of the proposed

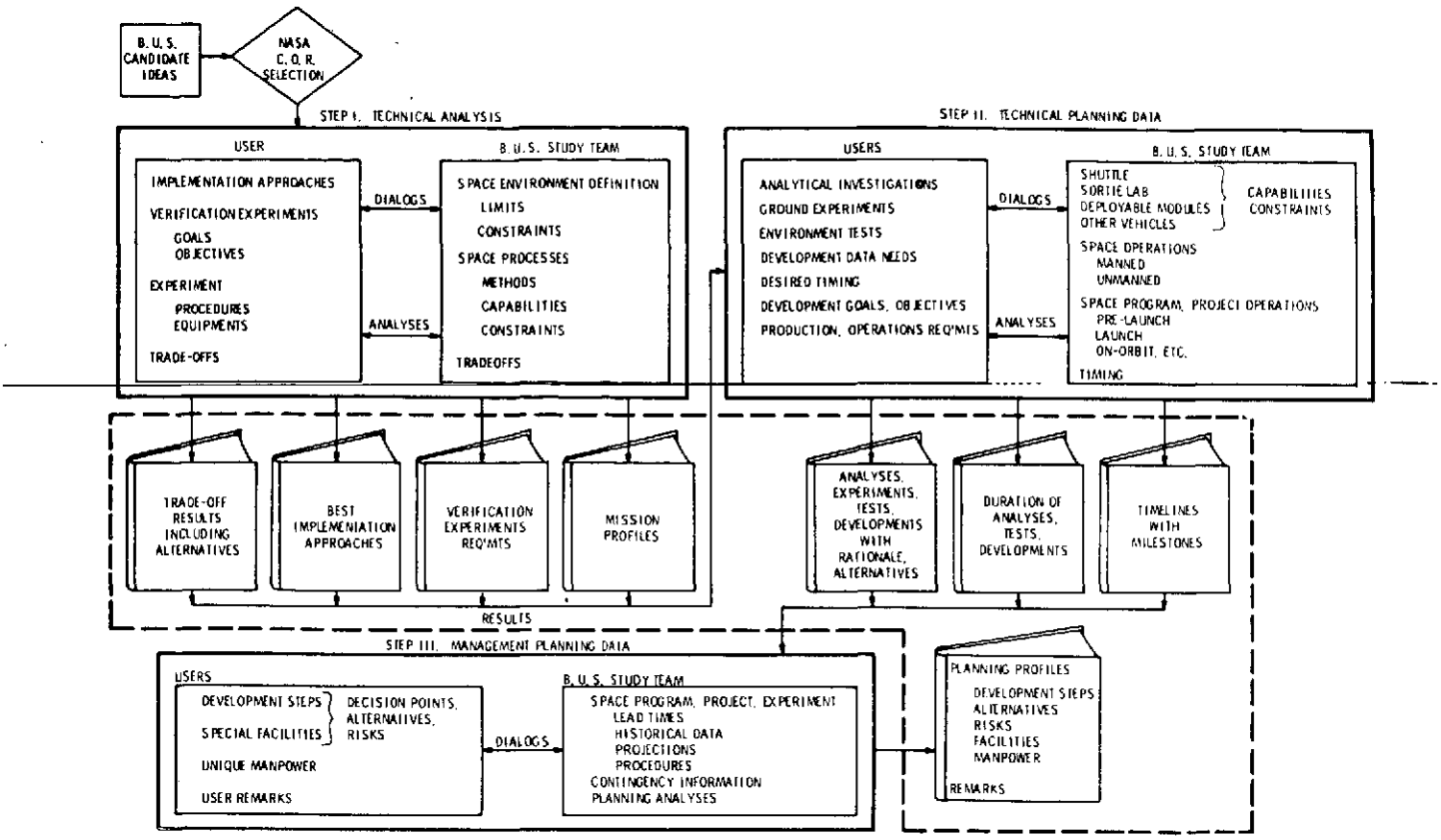

Figure II-1. Study Approach

study. The figure depicts the process by which the Study Team combined User-generated approaches and verification experiment requirements for implementing the selected Ideas with the methods, capabilities, limitations, and constraints inherent in space activities. Through that process, supplemented by analyses, as required, and supported by tradeoffs of alternatives, the participants derived realistic implementation approaches and the requirements for verification experiments. These results, in turn, enabled the Study Team to design the experiment mission profiles, and to suggest suitable types of vehicles for carrying out such missions.

Step II was carried out by a similar process. The figure shows that the Users' background in developing and planning the production, or operation of their own

products, processes and services was constrained by the unique characteristics of space "test beds", space programs and operations, as well as by key scheduling data. The Study Team utilized this step to define the implementation of each selected Idea by tabulation and timing of the major technical steps that must be accomplished in order to evolve from the present concepts to the eventual space products. As a result, the Study Team was able to construct timelines, with milestones, which formed the basis for management planning data in Step III.

Step III was organized to round out the planning information by providing an insight into the management-oriented aspects of implementing the selected Ideas. To encourage the important User input to this aspect of the Study, each User was free to utilize his own methodology for providing such planning data. The Study Team supplied background data to the various Users based on GE experience in space programs and projects so that there was uniformity in assumptions and baseline information. The Study Team also provided any necessary projections of that data, and provided definition of contingencies to be accounted for. Further, the Study Team analyzed the User results to formulate the planning profiles for each selected Idea. Dialogs were maintained throughout this step to provide the data exchange necessary for its successful accomplishment.

II.2 STUDY TASKS

Specific "packages" of work to be accomplished in accordance with the logic described above were documented in the form of Tasks, summarized in Figures II-2, II-3, II-4 and II-5. Each Task was performed for each of the four products under study.

II.2.1 TASK 1.0 - DEFINITION OF BEST IMPLEMENTATION APPROACH (Figure II-2)

Task 1 was aimed at selecting, from various alternative ways of producing each of the products under study, the one approach which was judged to be "best".

Each User, first, generated conceptual approaches for implementing each selected Idea. Definition of approaches was carried out in a combined Study Team/User effort to sufficient depth to enable comparison of approaches in a subsequent subtask. Definitions included, but were not limited to, the key environmental and process conditions required and/or created by the approach; major functions or process steps required in the approach; major operations required to support the approach; unique requirements for equipment, materials, volume, size, etc.; time frames for needs, availabilities; measures of values, costs, investments, etc.

TASK 1.0 DEFINITION OF BEST IMPLEMENTATION APPROACH

1.1 DEFINITION OF CANDIDATE APPROACHES

CONCEPTUAL METHODS, KEY CONDITIONS, MAJOR FUNCTIONS AND OPERATIONS, DRIVING REQUIREMENTS, TIMING

1.2 SELECTION OF BEST APPROACH

COMPARISON OF APPROACHES, TRADE-OFFS; CRITERIA: STRINGENCY OF CONDITIONS, COMPLEXITY OF FUNCTIONS AND OPERATIONS, POSSIBILITIES OF AUTOMATION, RELATIVE SIZE OF SPACE-BORNE EQUIPMENT, TYPE AND RELATIVE MAGNITUDE OF SUPPORT UTILITIES, TYPE AND RELATIVE MAGNITUDE OF RAW MATERIALS AND WASTE, TIMING AND AVAILABILITY VS NEED, QUALITY OF RESULT, RELATIVE PROGRAM COST

1.3 DEFINITION OF BEST APPROACH

ANALYZE BEST APPROACH FOR MAGNITUDES AND TOLERANCES OF PHENOMENA INVOLVED, FUNCTIONS AND SEQUENCES, PROCESS STEPS, INTERIM AND FINAL RESULTS

Figure II-2. Task 1.0

Next, the Study Team, with the identified Users, established a set of criteria for comparison of the alternative approaches. Criteria included, but were not limited to, level of tolerances of required environmental conditions required, precision of required performance factors, duration and precision of timing,

complexity of process functions, complexity of support operations, degree of automation feasible, relative size and weight of space-borne equipment; relative magnitude and varieties of support utilities and ground support services, relative magnitude and varieties of raw materials and waste products, relationships of needs for product, process or service to potential availabilities, relative quality of results from identified approach, relative cost of identified approach, etc.

We then compared identified approaches for each Idea against the above criteria, performing tradeoff studies where necessary to such comparison, and selected the best resulting approach.

To complete this Task, the Study Team/Users analyzed each selected best approach for information on its technical elements. Typically, these analyses provided data on such areas as specific phenomena involved in the specific techniques of the approach as well as their magnitude and tolerances, the functions that must occur and their sequence in order for the techniques to be successful, the specific steps (and sequences) of processes, the magnitude and quality of required results from each function, technique and process, etc.

II.2.2 TASK 2.0 - DEFINITION OF REQUIREMENTS FOR EXPERIMENTS TO VERIFY SELECTED APPROACH (Figure II-3)

Task 2 had the objective of defining the need for experiments to obtain the phenomenology, technology, and evolutionary data necessary to orderly development of the selected "best" approach.

This Task was initiated by review of each element of each best approach for the phenomena, techniques, process steps and results, as well as for the key relationships, whose successful accomplishment would contribute to the high probability

of successful development/production of the desired end product. These elements were then evaluated for their specific contributions; the sensitivity of the end product to each element, and the availability of alternatives. From such evaluation, we identified those elements contributing most to the end result, those to which the end results were most sensitive, and those for which limited alternatives existed.

In the next key effort, the Users established baseline definition of present and projected state-of-the-art in each technical area represented by the critical elements, using their experience in their specialized areas; and the Space Division Study Team performed an analogous definition, utilizing space applications data of current and planned programs documented in in-house and NASA-MSFC reports. We then analyzed specific critical elements of each best approach against the baseline to determine where specific "gaps" or weak points in available state-of-the-art would need "filling" in order to assure that each best approach could be implemented.

Supported by the Users, the Study Team next developed rationales for experiments (ground-, air-, or space-based) which could eliminate identified deficiencies in state-of-the-art baseline. For such experiments, we then generated definitive data (goals, objectives, estimated key procedures/equipments, timing, etc,) which established the requirements of those experiments.

II.2.3 TASK 3.0 - DESIGN OF MISSION PROFILES FOR EXPERIMENTS (Figure II-3)

Task 3 required the development of realistic sequences and timing of activities to carry out the above identified experiments. The Users/Study Team utilized the experiment requirements from Task 2 to construct, for each experiment, the sequence of operations and events that must occur in order for that experiment

TASK 2.0 DEFINITION OF REQUIREMENTS OF EXPERIMENTS TO VERIFY SELECTED APPROACH

2.1 IDENTIFICATION OF CRITICAL ELEMENTS IN SELECTED APPROACH

EXTRACT PHENOMENA, TECHNIQUES, PROCESS STEPS, RESULTS CRITICAL TO SUCCESS

2.2 ANALYSIS OF CRITICAL ELEMENTS

ANALYZE ELEMENTS AGAINST PRESENT KNOWLEDGE, DETERMINE STATE-OF-ART GAPS

2.3 DEFINITION OF EXPERIMENT REQUIREMENTS

GENERATE GOALS, OBJECTIVES, KEY PROCEDURES, MAJOR EQUIPMENTS REQUIRED TO FILL GAPS; CONSTRUCT RATIONALE ON ROLE OF EXPERIMENTS

TASK 3.0 DESIGN OF MISSION PROFILES FOR EXPERIMENTS

CONSTRUCT REALISTIC PROFILE OF TIMING, OPERATIONS, KEY SUPPORT, ETC.

Figure II-3. Tasks 2.0 and 3.0

to be successfully concluded. Based on past experience, analogous projects, and the above requirements, we assembled the schedule of the logical series and parallel operations for the actual experiment itself and the support activities necessary to its accomplishment into a mission profile for each experiment.

II.2.4 TASK 4.0 - ESTABLISHMENT OF TIMELINES AND MILESTONES (Figure II-4)

For planning purposes, it was important to develop realistic sequences and timing of Research and Development programs and events required to achieve production of the products identified in the Ideas selected for this Study.

The Study Team and Users, therefore, proceeded to identify, and estimate the duration of, analytical, experimental, design, test, fabrication, and operational

TASK 4.0 ESTABLISHMENT OF TIMELINES AND MILESTONES

4.1 REQUIRED RESEARCH AND DEVELOPMENT PROGRAMS

TABULATE SEQUENCE OF APPLIED R&D STEPS REQUIRED TO ACHIEVE PRODUCTION OPERATION. TYPICALLY, CONSIDER ANALYSIS, GROUND LAB TESTS AND EXPERIMENTS, AIRCRAFT FLIGHTS, ROCKET FLIGHTS, AUTOMATED SPACECRAFT, SHUTTLE FLIGHTS, SIMULATIONS, MATERIAL TESTS, ENGINEERING TESTS, SUPPORT EQUIPMENT DEVELOPMENT, PRE- AND POST-FLIGHT ACTIVITIES, TRAINING. ESTIMATE DURATIONS OF THESE

4.2 TIMELINES AND MILESTONES

ASSEMBLE TIMELINES FROM THE ABOVE FOR CONCEPT TO PRODUCTION OPERATION. SHUTTLE FLIGHT DATE OF 1982 FOR LATTER, WORK BACK TO FORMER. ACCOUNT FOR REALISTIC ALTERNATIVES IN TIMELINE. INDICATE SINGULAR POINTS AS MILESTONES

Figure II-4. Task 4.0

steps which must be performed to evolve each of the concepts represented by the selected Ideas into pilot plant production or operation. These steps were developed as logical applied research and development tasks that would be required in order to develop each approach from concept to pilot plant production/ operation. The Users' ground-based experience was integrated with the Study Team space program experience to provide the content of those tasks, and to estimate the duration of each step. As a guideline, the following tasks were considered for inclusion in each sequence:

- Analytical Investigations
- Experiments and Tests in Ground Laboratories
  - Drop Towers
  - Zero "G" Aircraft Flights
  - Sub-Orbital Rockets
  - Automated Satellites
  - Shuttle

- Environment Simulations and Tests
- Material Experiments and Tests
- Piece, Part, Component, Subsystem, System Tests
- Preflight Activities
- Post-Flight Activities
- User Process Training

In this activity, we accounted for applicable alternative program steps, considering both those alternatives which could not be resolved at this time due to lack of data, and those which could be chosen in the event a primary step would fail to provide successful results.

All the program steps were then assembled into a timed sequence, constrained to a 1982 shuttle pilot plant/operations goal, with logically adjusted sequences of steps and their durations to achieve a rational schedule. Considerable effort was required here to assemble timelines which were realistic from both User and space program viewpoints, considering both parallel tasks, where logical, and alternative tasks, where risks were adjudged high. Milestones were noted on the timeline for key tasks and significant singular events which impose driving requirements on schedules.

II.2.5 TASK 5.0 - FORMULATION OF PLANNING PROFILE (Figure II-5)

Task 5 was directed at providing a time-phased tabulation of key management information requirements; decisions, risks, alternatives, special facilities, unique manpower, etc. for implementing the above-generated programs for each selected Idea.

With the participating Users, we analyzed the foregoing derived programs to determine the alternatives faced by the implementer, and the specific decisions

TASK 5.0 FORMULATION OF PLANNING PROFILE

5.1 IDENTIFICATION OF DECISIONS

FROM ABOVE DATA, TABULATE DECISIONS REQUIRED PRIOR TO EACH DEVELOPMENT STEP. ESTIMATE SUCCESS PROBABILITIES/POSSIBILITIES OF EXPERIMENTS/TESTS. AT THESE POINTS IDENTIFY DECISIONS, ALTERNATIVES, RISKS

5.2 DEVELOPMENT OF DECISION TREE

COMBINE ABOVE INTO SINGLE, TIME-PHASED PLOT

5.3 OTHER CONSIDERATIONS

IDENTIFY SPECIAL FACILITIES, UNIQUE MANPOWER REQUIREMENTS IN PARALLEL TIME-PHASED TABULATION

Figure II-5. Task 5.0

required, as well as to estimate their concomitant risks and the lead times required between decision and action. Such decisions were tabulated together with the alternatives to be considered. Note was made of "trigger points" for decisions, the estimated risks associated with these alternatives, and the required lead times. It was important to examine each program step for relationship to preceding, parallel, and succeeding steps, as well as for its impact on the total program in order to uncover such decisions and risks.

As a final effort in the Study, a time-phased plot of the identified decisions was assembled, with flows indicating the prerequisite and successive relationships among decisions. Timing followed the time frame of the previously constructed timelines, and lead times were reviewed (and modified, where required)

against User experience and space program experience. All plotted decisions were documented with appropriate alternatives, risks and preferences identified by the Users.

SECTION III

STUDY RESULTS

This section of the Technical Report provides the specific information developed during the Study in response to the Tasks defined in Section II, Method of the Study.

Accordingly, the Study Results are organized herein in a sequence which parallels those Tasks.

- The first four subsections cover selection of the best implementation approach for each of the four Ideas under study.
- The next four subsections cover definition of experiment requirements for the selected implementation approaches.
- A subsection then follows on Mission Profiles for the defined experiments.
- A subsequent subsection presents the program timelines and milestones for developing the best implementation approaches.
- The final subsection deals with the decision flows involved in carrying out the above programs.

III.1 SELECTION OF BEST APPROACH FOR SEPARATION OF ISOENZYMES

Enzymes are proteins which catalyze the majority of chemical reactions in all living organisms. Enzymes play such important and specific roles in metabolic processes that they are vital to the existence and sustaining of life. A slight deficiency or abundance of a single enzyme may be manifested in severe metabolic disorders and diseases, e.g., genetic deficiency of the enzyme hexosaminidase causes the deadly Tay-Sach's disease.

Recent advances in protein isolation techniques have shown that enzymes can exist in slightly different molecular forms. These alternate forms of an

enzyme, which differ slightly in physiochemical properties are called isoenzymes. At present there are approximately one hundred of the nearly 2,000 enzymes which are known to have such forms.

Structurally, enzymes and hence isoenzymes, are globular proteins. Being protein molecules, differences in isoenzyme structures can arise from slight and subtle variations in their primary, secondary, tertiary and quaternary structures. The forces holding these structures together may be weak interaction forces between the groups on the protein chain and gentle means of separating must be used to avoid denaturation, that is, modifying those structures, during the process of separation.

Each enzyme is viewed as having a discrete, folded conformation which helps the specific binding of the substrate at its active site. This is necessary for the enzyme reaction (lock and key concept of enzyme action).

The forces stabilizing this folded conformation are weak and hence enzymes are rather sensitive proteins. These mild forces of aggregation are very easily disturbed by heat, chemical agents and electrical potentials as in conventional electrophoresis at high voltage. These conditions cause the unfolding of the enzyme structure (denaturation) resulting in loss of enzyme activity.

An interesting aspect of isoenzymes has recently come from their clinical value as indicators of abnormal or diseased tissue or organ states. It has been well documented that such abnormalities as myocardial infarctions and muscular dystrophy, cancer, etc. are accompanied by elevated levels of certain characteristic isoenzymes of lactate dehydrogenase and creatine kinase. Figure III-1 summarizes some of the clinical implications of isoenzymes.

ABNORMALITY	KEY ISOENZYME PATTERN ALTERATION COMPARED TO NORMAL
MYOCARDIAL INFARCTION	LACTATE DEHYDROGENASE, CREATINE KINASE
LIVER DISEASE	LACTATE DEHYDROGENASE, ARYLAMIDASE (LEUCINE AMINO PEPTIDASE)
MUSCULAR DYSTROPHY	LACTATE DEHYDROGENASE, CREATINE KINASE
RENAL DISEASE	LACTATE DEHYDROGENASE, ALKALINE PHOSPHATASE
NERVOUS SYSTEM DISORDERS	LACTATE DEHYDROGENASE
CEREBRAL INFARCTION	CREATINE KINASE
CANCER	LACTATE DEHYDROGENASE, GLUCOSE-6-PHOSPHATE DEHYDROGENASE, PHOSPHYGLUCONATE DEHYDROGENASE
GLYCOGEN STORAGE DISEASE	GLYCOGEN PHOSPHORYLASE
INFECTIOUS DISORDERS	CATALASE

Figure III-1. Summary of Some Clinical Applications of Isoenzymes

The most commonly and successfully employed isoenzyme isolation technique has been conventional small pore gel electrophoresis. With the development of cellulose acetate strips, starch and polyacrylamide gels, and isoelectric focusing, the resolving power has been progessively improved. Therefore, what was originally thought to be a homogeneous protein with cellulose acetate electrophoresis was shown to be quite heterogeneous on starch gel electrophoresis and so on. For example, L-amino oxidase previously thought to exist in three forms, as determined by electrophoresis was shown to contain 18 isoenzymes by isoelectric focusing technique. With each new refinement in separation technique, significant new advances has been made in our understanding of biological, and consequently, medical phenomena.

At present, the major obstacles in the full exploitation of isoenzymes as clinical diagnostic tools are:

(1) The difficulty of their isolation under delicate conditions in sufficient quantities of pure form for detailed study

(2) A convenient means for detecting very small levels of the "diseased or malfunction" indicating isoenzyme.

It is apparent, therefore, that any isolation method which could readily provide reasonable yields of high purity isoenzymes would have a large impact on the early clinical diagnosis of various diseases and also go far in providing tools for further elucidation of the basic cell process.

III.1.1 PRESENT METHODS OF SEPARATION OF ISOENZYMES

(1) Electrophoresis: paper; cellulose acetate; gel-starch, polyacrylamide, agar; column-starch, cellulose

(2) Isoelectric Focusing: sucrose gradient, polyacrylamide gel

(3) Chromatographic Techniques: ion exchange; gel filtration; hydroxyapatite

The problems with present methods of isoenzyme separation may be summarized as follows.

III.1.1.1 Electrophoresis and Isoelectric Focussing

(1) Sedimentation

(2) Convective mixing of the separated bands (medium)

(3) Limited pore size of the gel medium thus excluding very large molecules

(4) Thermal effects due to high electrical resistance of the small pore gel medium and need for high voltage

(5) Absorption on the medium. Small pore gels are relatively high solids with relatively high internal surfaces

III.1.1.2 Chromatographic Techniques

(1) Limited pore size (gel filtration)

(2) Absorption

(3) Separation based only on molecular size (gel filtration)

(4) Denaturization of enzyme due to surface action

III,1,1.3 Projected Ground Separation Techniques

Electrophoresis under rotational motion, developed by Dr. Alexander Kolin of UCLA, was an attempt to obviate the gravitational effect on the convective mixing. In this method the convection of the fluid medium was minimized by rotational motion of the medium by application of perpendicular magnetic and electrical fields, whereby charged particles migrate in helical paths of different pitches depending on their net charge. While this method could be applied to separate large particles such as kidney cells, it is the opinion of such authorities as Dr. G. Barlow of Abbott Laboratories, Chicago, that this method is not likely to be useful for separation of smaller particles such as isoenzymes.

III.1.2 APPROACHES TO SEPARATION OF ISOENZYMES IN ORBIT

Since some of the problems listed for present separation techniques have their basis in the one "G" environment of Earth, it may be worthwhile to consider utilizing such techniques, or modifications thereof, in space. Consideration has been given to the generic techniques, listed in Figure III-2.

1.	LARGE-PORE GEL ELECTROPHORESIS
2.	LARGE-PORE GEL ISOELECTRIC FOCUSSING
3.	SEPARATION BY OTHER WEAK PROPELLING FORCES, MAGNETIC FLUX ULTRASOUND, FRACTIONAL GRAVITATIONAL FORCE USING LARGE-PORE GELS. THESE SEVERAL EXAMPLES OF FIELD FORCES ARE CAPABLE OF CONTROLLED ATTENUATION AND ARE THOUGHT TO BE GENTLE ENOUGH TO AVOID THE DENATURATION OF THE NATIVE PROTEINS.
4.	SMALL PORE GEL ELECTROPHORESIS
5.	FREE FLOW ELECTROPHORESIS
6.	COLUMN CHROMATOGRAPHY
7.	THIN LAYER CHROMATOGRAPHY

Figure III-2. Potential In-Space Separation Techniques

A generic representation of the space separation approaches is shown in Figure III-3.

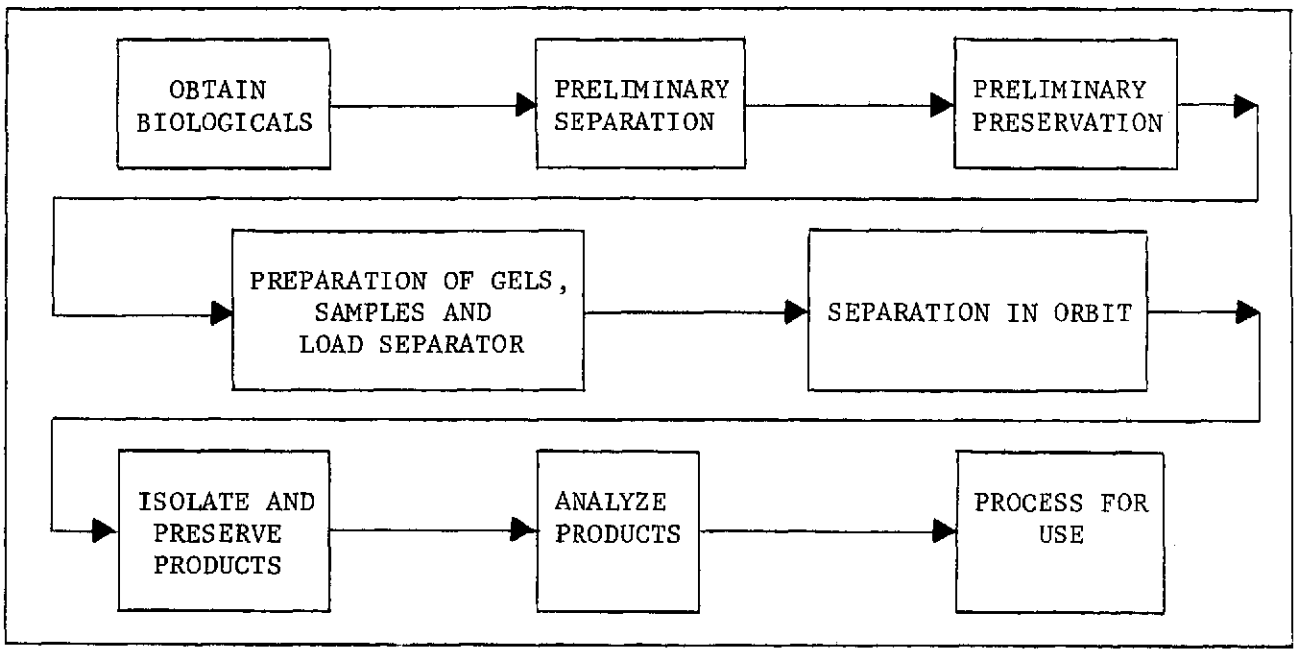

Figure III-3. Isoenzyme Processing

The process steps, and their alternatives, for the approaches represented in Figure III-3 are discussed below.

III.1.2.1 Obtaining Biologicals

Source biologicals may be obtained from serum, cell or micro-organism culture, or tissue or organ extract. As shown in Figure III-4, these are generally available from a number of sources.

III.1.2.2 Preliminary Separation and Preservation (Figure III-5)

III.1.2.2.1 Initial purification is usually performed by:

(a) Acetone Precipitation

(b) Sodium Sulfate Precipitation, etc.

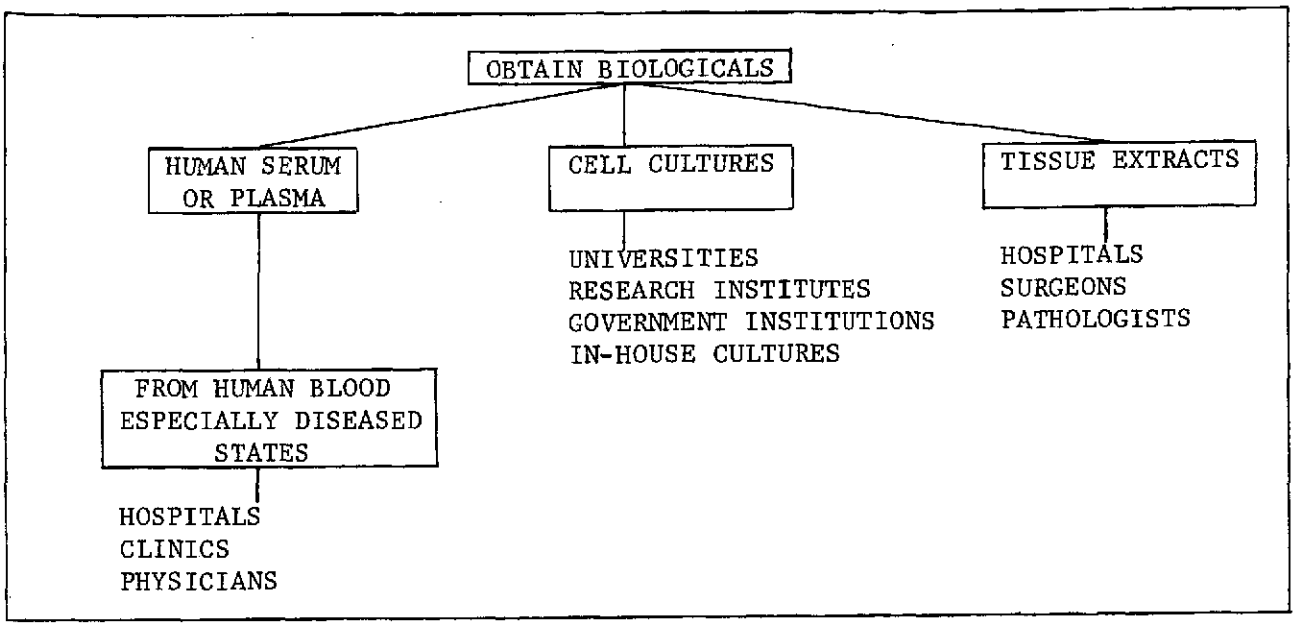

Figure III-4. Obtain Biologicals

III.1.2.2.2 Secondary purification may be performed by:

(a) Column Chromatography on Siphadex (gel filtration), Ion Exchange Resins, and other materials

(b) Preliminary Electrophoretic Separation

(c) Selective Denaturization of Undesirable Protein

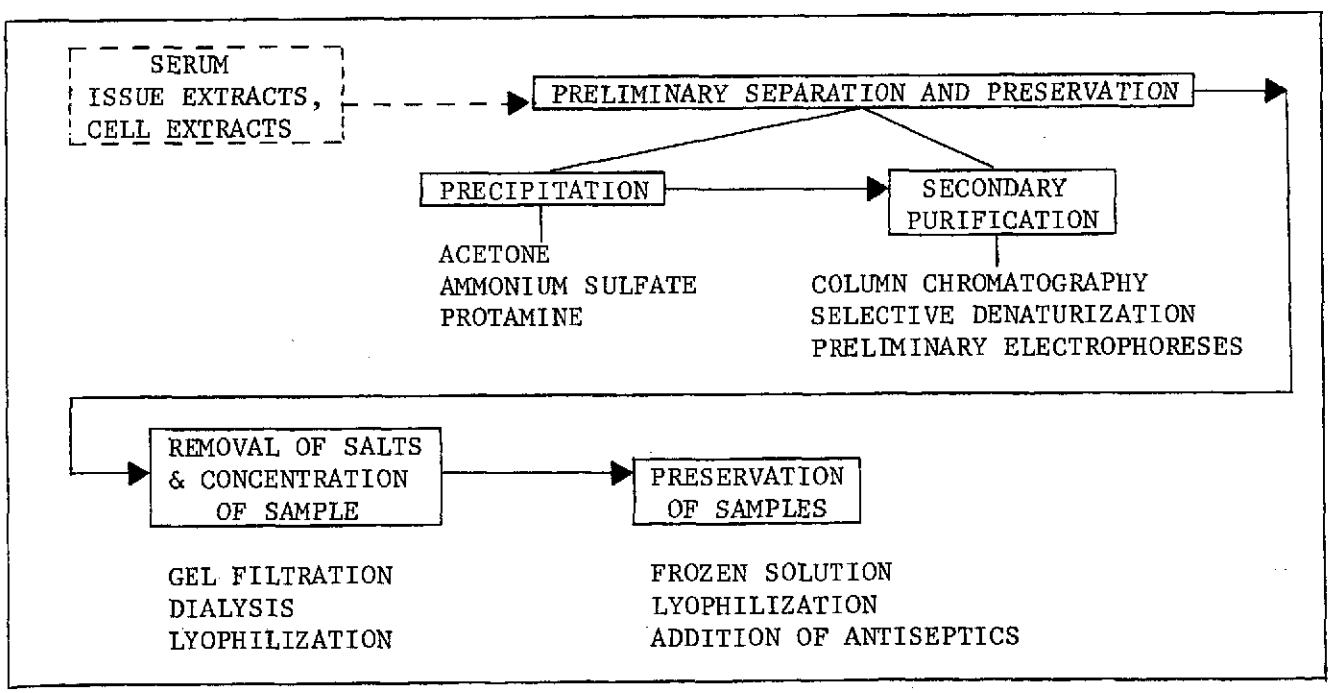

Figure III-5. Preliminary Separation and Preservation

III.1.2.2.3 Removal of small molecular weight material and concentration of sample is performed by:

(a) Gel Filtration

(b) Dialysis

(c) Lyophilization - Freeze Drying

III.1.2.2.4 Storage of sample by:

(a) Freezing the Solution

(b) Lyophilization to a Dry Powder

(c) Addition of Antiseptics

III.1.2.3 Preparation of Gels, Samples, Loading of Separator (Figure III-6)

(a) Among the early decisions that must be made in order to proceed with this Idea is that of gel composition, density, etc. This chart merely lists some of the alternatives involved which will devolve from these decisions.

(b) A key alternative to be faced is whether to form and load such gels prior to launch, or to perform those function in space.

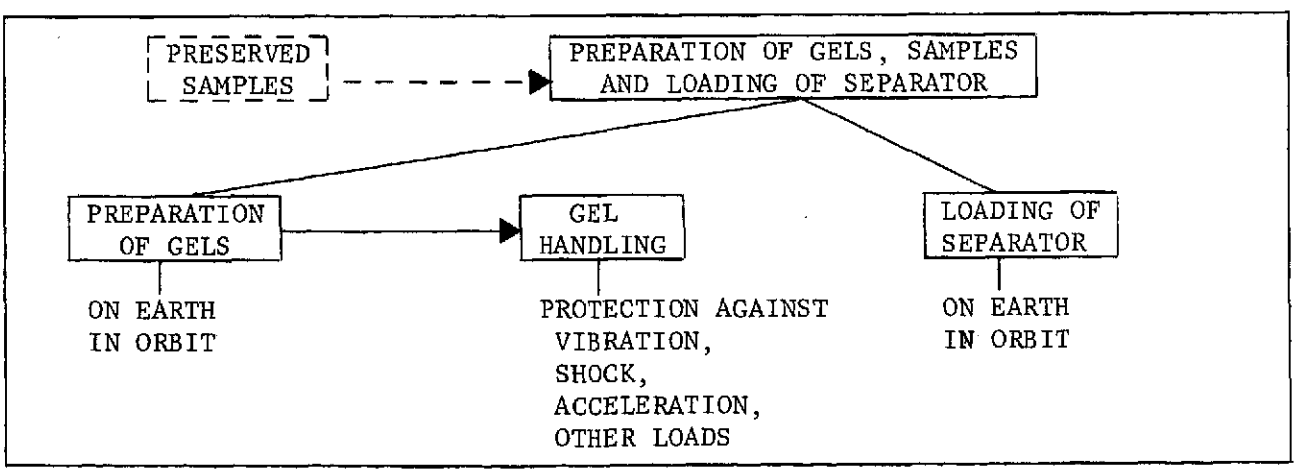

Figure III-6. Preparation of Gels, Samples, and Loading of Separator

III.1.2.4 Separation Methods (Figure III-7)

III.1.2.4.1 Electrophoresis

Electrophoresis is based on the differential mobility of ionized particles through a medium under the influence of a voltage gradient. Mobility depends on net charge and on molecular size (if there is a sieving effect on small pore gels). The sieving effect may improve resolution at the cost of distortion and frictional resistance of the particles as they pass through the pores. This may cause denaturation of sensitive components.

III.1.2.4.1.1 Medium. May be free flow (liquid) or a buffer solution in paper, cellulose, acetate, starch, agar, or polyacrylamide (small pore or large pore).

III.1.2.4.1.2 Gel. A gel, if used, is supported by glass or plastic and is either in the form of a slab or a cylindrical column. A free flow system will not require a gel.

III.1.2.4.1.3 Buffers. Each end of the gel is in contact with a buffer solution which may be the same at each end (continuous system) or different (discontinuous). In general, discontinuous systems give better resolution (sharper bands). An electrode is present in each buffer chamber.

III.1.2.4.1.4 Energy Supply. A circuit which produces DC current at constant voltage and/or constant current is required. Voltages up to 1000 V and currents up to 100 mA may be needed.

III.1.2.4.1.5 Separation Operation. The sample is introduced as a concentrated solution on top of the gel, or contained in a "sample gel" on top of a running-gel. A "stacking gel" is usually needed to compress the sample into a very thin band

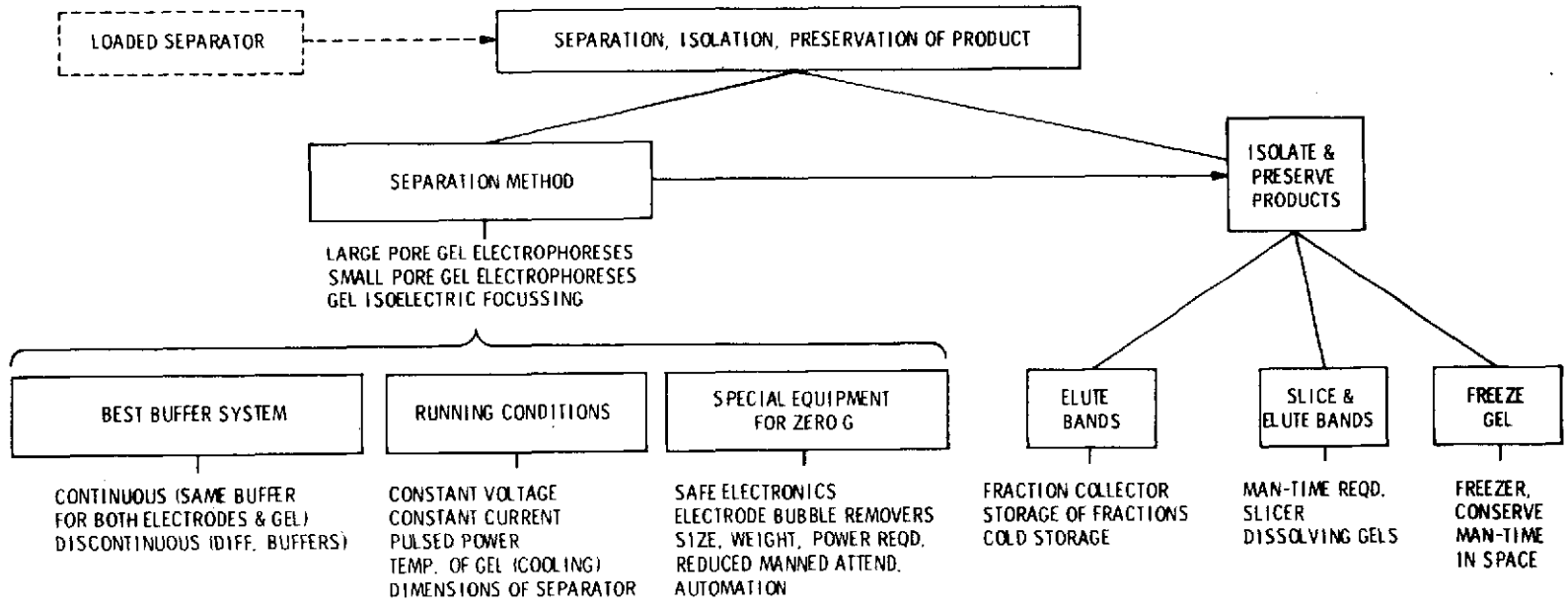

Figure III-7. Separation, Isolation, Preservation of Product

before it enters the separation gel (disc-electrophoresis). The sharpness and uniformity of the sample band is critical for superior resolution and this may be a problem for in-orbit operations. A free-flow apparatus also requires a sharp band of sample for good resolution, but a sharp band is more difficult to attain in a free flow system.

After addition of the sample, a current is applied and the ionized particles move through the medium separating into component bands. The resistance of the medium causes heating, and cooling of the medium may be necessary to prevent heat denaturation of the biological components. As the bands progressively separate, they are subjected to continuous disruptive forces which broaden the bands and reduce resolution. Free flow is especially sensitive to thermal convection, vibrations and jarring. The disruptive forces limit the time and path length of a run. Zero gravity conditions may make possible runs of longer duration, either by lower voltage gradients (less heating) or longer path lengths, and thus may yield improved resolution of closely spaced bands.

III.1.2.4.1.6 Isolation of Separated Components. Free-Flow devices can be complicated multi-chambered systems which allow the "tapping" of bands during or immediately after a run. It is doubtful that a completed run can be stored for later isolation of component bands.

With gels, the separated bands can be eluted from the end of the gel and collected in small volumes (fractions). There is the difficulty of "remixing" of bands as they are collected.

Another approach is to run only until the bands are separated, followed by removal of the gel from its support, slicing into thin cross-sections and eluting any material from each slice separately. The advantages are less running time,

and no remixing. The disadvantages are longer manipulation time, and difficulty of eluting. The eluting problem has been partially solved by the invention of gels which dissolve by chemical or physical treatment, allowing the material in the gel to be easily recovered. More work is needed on "dissolving" gels.

The best alternative, if possible, is to merely freeze the gel after a run, for further isolation and elution on earth. Tests must be performed on loss of resolution in frozen storage.

III.1.2.4.2 Isoelectric Focussing

The methods and equipment for isoelectric focussing are very similar to those of electrophoresis with the following modifications. An ampholyte mixture is incorporated into the gel, and the sample may also be incorporated into the monomer solution before polymerization, or may be added as a solution to the top of the gel. The quality of separation is not dependent on having a sharp band of sample, so that sample loading is not a critical factor (as it is in gel electrophoresis), and no stacking gel is required.

When a voltage gradient is applied, the ampholtye mixture is ordered in the gel to form a smooth pH gradient. The ultimate resolution of the products is strongly dependent on the formation and stability of this pH gradient. A free-flow apparatus will not maintain a good gradient because of convection, diffusion, and will not be useful for isoelectric focussing. After establishment of the pH gradient, the biological components migrate to the pH in the gel equivalent to their own PI (isoelectric point).

The advantages of isoelectric focussing are very high resolution and no distortion or frictional resistance (a large pore gel is used). The disadvantages are somewhat longer running time and the denaturation of certain proteins when held at their PI.

III.1.2.4.2.1 Isolation of Separated Components. Elution of the bands from the end of the gel is not possible in isoelectric focussing. The gel can be sliced into cross-sections as discussed for electrophoresis, can be frozen for later processing as discussed, or the entire gel can be "dissolved" by chemical or physical treatment, and the liquid column eluted in fractions.

III.1.2.4.3 Other Separating Techniques

III.1.2.4.3.1 Column Chromatography. Ion exchange, gel filtration, hydroxyapatite. A solution of biological compounds flows past a solid phase which retards the movement of each component by differential adsorption or ionic binding. Column chromatography is well adapted to preparative scale separations, but resolution is very poor, and there is no obvious zero-gravity advantage.

III.1.2.4.3.2 Vapor Phase Chromatography. V.P. chromatography is unsuited to separation of high molecular weight, non-volatile biological compounds.

III.1.2.4.3.3 Thin Layer Chromatography. Similar to column chromatography but with better resolution. However, there is no zero gravity advantage, and the loading capacity is small.

III.1.2.4.3.4 Migration of Components Under the Influence of Weak Forces. Limiting velocities of micron-sized particles moving in air have been calculated for forces induced by magnetic fields, electromagnetic radiation pressure, resonant radiation pressure, and sound pressure. The values range from 10^{-5} to 3×10^{-3} cm/sec. But for smaller particles in aqueous solutions, the velocities would be at least two orders of magnitude lower, making these forces generally unsuitable for the separations proposed. Also, so little is known about the devices needed, that a major project is required to learn the basic properties of these separation methods.

III.1.2.5 Analysis and Final Processing (Figure III-8)

III.1.2.5.1 Assay of Separated Components

The separated components must be assayed for purity and biological activity. The methods used will depend primarily on the particular systems to be studied. Methods usually include UV adsorbance, enzyme assays with synthetic or natural substrates, and crystallization.

III.1.2.5.2 Final Processing

The final processing will depend on end use. For individual diagnosis the isoenzyme itself may be desired, while for large scale diagnosis, where it is possible to produce viable, tolerable, approved anti-body solutions, such anti-bodies would be produced and stored.

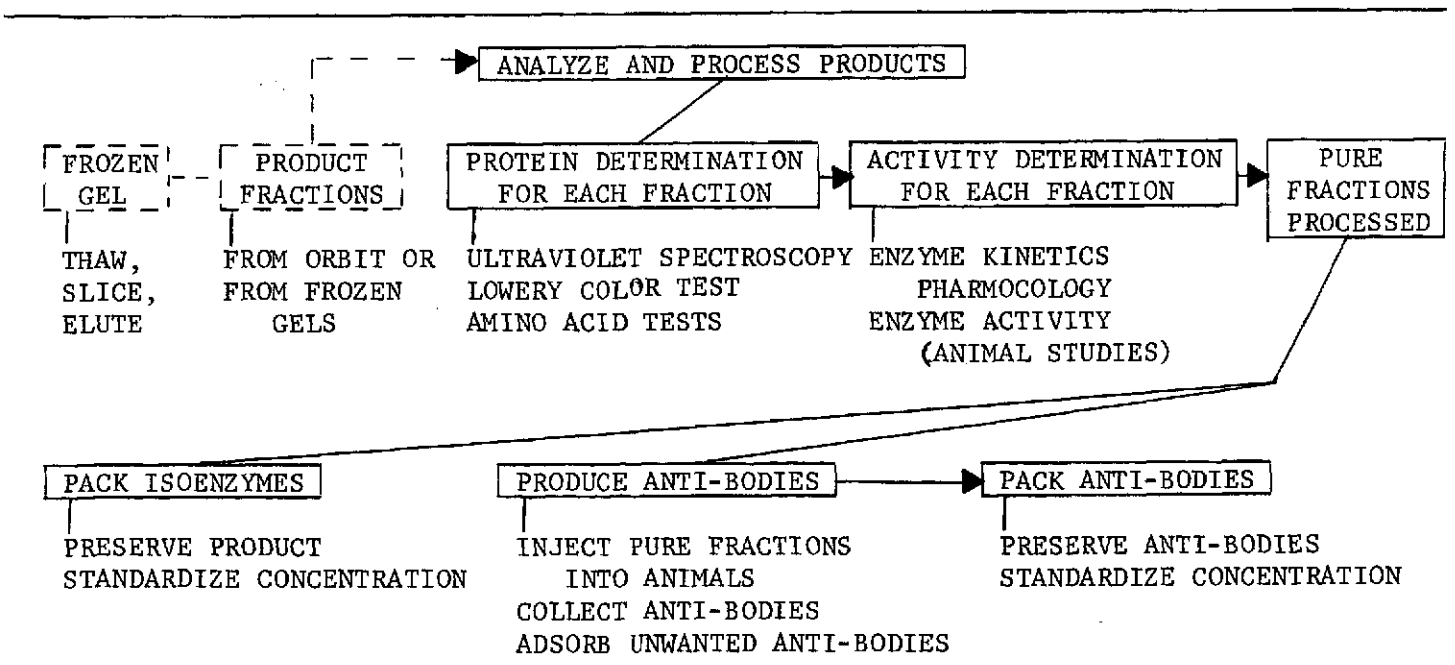

Figure III-8. Analysis of Products and Processing for Use

III.1.3 COMPARISON OF APPROACHES FOR SEPARATION OF ISOENZYMES

III.1.3.1 Definition of Criteria for Candidate Approaches

III.1.3.1.1 Resolving Power

The resolution of very similar isoenzymes is the basic requirement for obtaining pure products from space. It is essential to use the best methods, based on earth results. Of the methods discussed, only gel electrophoresis (large gel and small pore) and gel isoelectric focussing appear to have the resolving power necessary.

III.1.3.1.2 Advantage of Zero Gravity

Improvements in sedimentation, convective mixing and magnitude of required motive forces should accrue to some separation techniques under zero gravity conditions. This criterion would rule out chromatographic methods, since a liquid moving past a solid phase will be little affected by convection, etc. compared to the problems of turbulence and "channeling" which disrupt chromatographic separations.

III.1.3.1.3 Present Knowledge of the System

A method should be chosen which has been examined to a reasonable degree on earth, and is known to give usable results. This criterion would rule out separations based on the use of "weak forces".

III.1.3.1.4 Stability of Components

The method must be gentle enough so that desired products are not denatured or aggregate structures broken apart.

III.1.3.1.5 Relative Reliability of Equipment

The type of equipment anticipated must be resistant to launch and reentry conditions, must be designed to work properly under zero graviey (i.e., closed buffer tanks), and should be relatively simple to operate.

III.1.3.1.6 Amount of Manipulation

As many of the processes, such as formation of the gels, sample loading, product isolation, as is possible should be done on earth leaving only the separation operation for zero gravity if possible.

III.1.3.1.7 Size, Weight and Power Requirements

For electrophoresis or isoelectro-focussing, similar equipment is used. In fact, both large and small pore gel electrophoresis and isoelectric-focussing can be done with the same equipment, thus gaining extra capability with no extra cost in equipment.

III.1.3.1.8 Duration of Separation Operation

The time for an electrophoretic or isoelectric-focussing run is on the order of 1 to 5 hours and requires little attention during this operation. Times for other methods are somewhat longer, 2 to 10 hours.

III.1.3.1.9 Amount of Earth Support

For all methods, the amount of time spent will be largely for the isolation and assay of the resulting products.

III.1.3.2 Comparison of the Alternative Approaches

III.1.3.2.1 Separation Approaches

The major step in the total process is the high specificity separation. When the above criteria are given weights, and the candidate separation approaches

are ranked with the most favorable approach given 10, and other approaches are ranked less, for each criterion, a weighted total can be calculated for each approach. Figure III-9 presents the matrix of separation alternatives versus criteria, as judged for relative worth by the User/Study Team. The three top approaches are seen to be gel electrophoresis (small pore), gel electrophoresis (large pore), and gel isoelectric-focussing.

This chart represents a simplified comparison of the alternatives noted for this step -- large- and small- pore gel electrophoresis, isoelectric-focussing, free flow electrophoresis, and use of other small forces.

Little work has been done with the last-listed alternative, thus that tabulated data has little meaning. Large pore gel electrophoresis, especially when combined with isoelectric-focussing appears to have significant advantages. Free flow electrophoresis has been listed, but is actually more applicable for other product goals. The isoenzymes covered in this Idea are under consideration for preparative utilization in small amounts (hundreds of milligrams) for diagnosis, whereas the larger volumes of products potentially achievable with free flow electrophoresis will likely find use in large scale treatment applications.

III.1.3.2.2 Approaches for the Other Process Steps

The selection of the best separation approach, in general, simplifies the selections from among the other, less critical process steps. Selections in these other steps are discussed below.

III.1.3.2.2.1 Sample Preparation will be the same for gel electrophoresis and gel isoelectric-focussing except that the sample should be more concentrated for gel electrophoresis because of the requirement of a sharp sample band.

CRITERION	WT. MULT.	GEL* ELECTROPHORESIS SMALL PORE	GEL* ELECTROPHORESIS LARGE PORE	ISOELECTRIC* FOCUSSING	FREE-FLOW ELECTROPHORESIS	COLUMN CHROMATOGRAPH	THIN LAYER CHROMATOGRAPH	OTHER WEAK FORCES
RESOLVING POWER	10	8	8	10	7	1	1	UNKNOWN
ADV. OF ZERO GRAVITY	10	8	10	10	10	1	1	10
PRESENT KNOWLEDGE OF SYSTEM	9	10	8	8	7	10	10	1
STABILITY OF COMPONENTS IN SYSTEM	9	7	10	8	7	4	4	UNKNOWN
RELIABILITY OF EQUIPMENT	6	5	5	5	5	10	10	1
AMOUNT OF MANIPULATION (AUTOMATION FACTOR)	6	6	6	5	10	8	10	6
SIZE, WT, AND POWER REQUIREMENTS	5	5	5	5	6	8	10	1
DURATION OF SEPARATION OPERATION	4	8	8	7	9	4	10	1
AMOUNT OF EARTH SUPPORT	2	8	8	8	10	10	10	1
WEIGHTED TOTALS	----	452	481	473	472	330	376	162

* CAN USE THE SAME EQUIPMENT

Figure III-9. Comparison of Isoenzyme Separation Methods

III.1.3.2.2.2 Gel Preparation. Small pore polyacrylamide gels have been used the longest for separation of proteins, particularly from serum. It had long been assumed that small pore gels gave superior resolutions because of molecular sieving, but recent work at Polysciences shows that large pore gels produced from polyethylene glycol diacrylate (PEGDA) or polyacrylamide cross-linked with GEPA gives better resolution of serum proteins.

For larger molecules, like RNA and DNA, large pore gels are required to obtain any substantial mobility.

Large pore gels are also best for isoelectric-focussing, since rapid mobility of components is a definite advantage, and the sieving effect of small pore gels a disadvantage. Suitable "dissolving" or "reversable" gels are presently being studied at Polyscience.

Gels would be best prepared on earth, if they are found able to stand launch conditions.

III.1.3.2.2.3 Sample Loading. Sample loading on gel isoelectric-focussing is much less critical than on gel electrophoresis. In fact, for gel isoelectric-focussing, the sample may be incorporated into the gel on earth. For gel electrophoresis, the sample would best be incorporated in a sample gel on earth, as it is difficult to layer a liquid sample on a gel in zero gravity.

III.1.3.2.2.4 Equipment. The electronic part is simple and adaptable to both gel isoelectric-focussing and gel electrophoresis, but the gel holder, buffer chambers, etc. must be designed specifically for space use. A refrigerator-cooling system may be needed to store samples and gels, and to cool the gel while running.

III.1.3.2.2.5 Fractionation Equipment. If the separated components must be separated in orbit, a device to automatically collect fractions and store them will be needed. Such devices have been developed but they are not too reliable and further development may have to be carried out. This is a problem for both gel isoelectric-focussing and gel electrophoresis.

III.1.4 DEFINITION OF SELECTED APPROACH TO SEPARATION OF ISOENZYMES

The resolving power of an electrophoretic system is limited by convective disturbances on the medium mediated by temperature variations within the medium, and by diffusion of the molecules of interest. The use of gels in place of free solution electrophoresis has improved resolution greatly by reducing both disturbing effects. However, convective disruption is still a cause of band broadening in large pore gel systems and more so in free flow systems. The use of zero gravity should substantially improve resolution in these special gel systems.

Large pore gels are superior to free electrophoresis in several other aspects. The sieving effect of the gel on the macromolecules provides a way of separating molecules on the basis of molecular volume in addition to the net charge differences which is the only mechanism operative in free flow. Gels can be prepared which contain covalently bound cationic or anonic charges, or specific moieties like coenzymes. The molecules passing through this modified gel are then separated on their relative affinities to the covalently bound groups. Gels also provide an opportunity to use the method of "stacking". That is, when the sample passes through an interface between two gels containing different buffers, the molecules are compressed into an extremely sharp band, which is then separated into very sharp bands of the individual molecule types, greatly improving resolution. It is interesting that the stacking phenomenon is almost independent

of sample size, and increased loads of sample do not result in broader bands. Gels also protect the separated bands from vibration, jarring, and shock and one mode of producing separated products in space is to store the gel after separation in orbit for isolation of the products on the ground - this option is not open to free electrophoresis.

Free flow electrophoresis, being a continuous system, has the possibility of producing larger quantities of materials, and being a free liquid system, allows particles of large size, like cells which cannot penetrate gels, to be separated. On the other hand, because of turbulence, variations in flow rate, and sample entrance into the system, we believe a free flow method cannot be refined to provide the very high resolution already achieved with our gel systems, and necessary to separate such microheterogenous systems as isoenzymes, polynucleotides, immunoglobulins, histones, cell organelles and others, all of which are of tremendous importance in medically related areas. Therefore, we believe the gel and free flow methods are not directly comparable since the particle types to be separated can be handled by one or the other method but not both methods.

Regarding the quantities of separated molecules needed, we intend to separate isoenzymes and other molecules peculiar to disease states and to use these biomolecules to hyperimmunize animals. The immunized animals produce anti-body which is then used to detect the presence of the abnormal molecules in clinical practice, either in a mass screening program, or to confirm a suspected diagnosis in selected patients.

In practice, a few milligrams of purified macromolecule is sufficient to immunize an animal like a goat or horse, and with occasional booster injections

of perhaps one milligram/3 weeks, about 20 ML of serum per week containing anti-body is obtained. Using radioactive or fluorescence immunoassay, this 20 ML of serum per week should be sufficient for about one thousand immunoassays. Therefore, as little as 10 MG of space product should provide for immunoassays on 1000 persons per week for several months. We believe that a preparative gel electrophoresis system in space could provide perhaps 100 MG, pure product per run, with each run requiring several hours. Therefore, a batch process is certainly sufficient for our needs.

New macromolecules are well known to appear in disease states. For example, the sera of patients with hepatoma (liver cancer) was shown to contain a phosphodiesterase isoenzyme not found in normal sera. However, it is critical to detect these abnormal molecules in the very early stages of disease in order to achieve a cure. In the earliest stages of hepatoma, the abnormal isoenzyme will be present in concentrations far too low to detect by ordinary enzyme techniques like chromagenic enzyme substrate, and only the tremendously sensitive radioimmunoassay or fluorescence immunoassay will be sufficient for the early diagnosis. This is why we must find these new isoenzymes using extremely high resolution methods better than now available, and make them available in a pure state in quantities of several hundred milligrams, for anti-body production.

The simplified diagram given in Figure III-3 for isoenzyme processing, when expanded to include all the reasonable alternatives is shown in Figure III-10. That figure also identifies the approach selected for this Study, and the location, ground or orbit, for each process step.

FOLDOUT FRAME 1

FOLDOUT FRAME 2

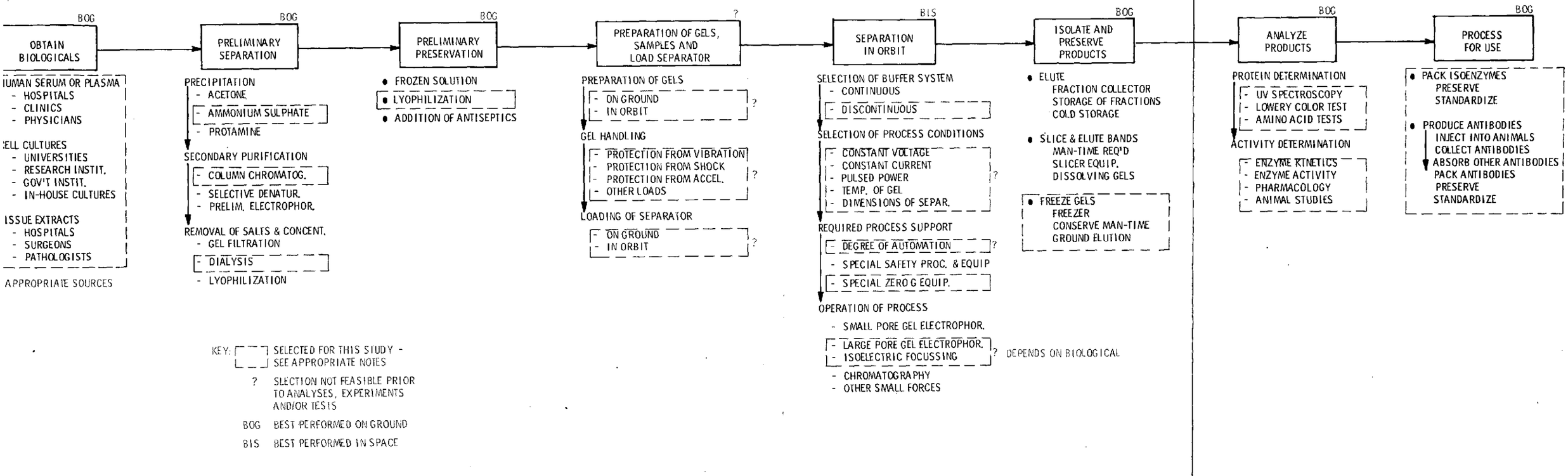

Figure III-10. Definition Of Best Implementation Approach For Separation of Isoenzymes

III.2 SELECTION OF BEST APPROACH FOR PROCESSING OF TRANSPARENT OXIDES

Design of optical instruments is frequently limited by available optical materials. Besides optical properties, thermal and chemical stability, as well as mechanical hardness are also desirable to enable operation in severe environments.

Specifically, new combinations of refractive index and dispersion, and extended transmission into the infrared and ultraviolet regions would be welcome. The need for materials possessing these properties has been noted in some detail by Augustyn and Alte[1] and the Materials Advisory Board of the National Academy of Sciences/National Academy of Engineering[2].

Typically, the properties sought for are:

Index of Refraction:	Approximately 2.0
Dispersion:	20
Thermal Stability:	Useable at temperatures up to 1000°C with stable properties to that temperature
Chemical Stability Optical Transmission Abrasion Resistance	At least as good as present silicate glasses

The oxides of aluminum, zirconium, and yttrium are recommended for initial processing. When quenched from the melt in spherules 100 - 800 μm diameter, all of these oxides have remained amorphous. Therefore, they seem like appropriate candidates for processing aimed at producing glasses in sizes large enough to be practical for components of optical systems.

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(1) Augustyn, W. H. and Alte, E. L., Contributors to report No. SN72-SA-0083, Study of the Production of Unique New Glasses, 12 June 1972.

(2) Ad Hoc Committee on Infrared Transmitting Materials, Materials Advisory Board, Division of Engineering, National Research Council, Publication MAB 243, July, 1968.

According to criteria formulated by Zachariasen[3] alumina and zirconia will not form glasses. But Sun[4], considering high bond strength as a requisite for glass formation, lists both alumina and zirconia as potential glass formers. Classifying oxides by their position in the periodic table, Winter[5] concludes that alumina is a potential glass former but that zirconia is not. Zarzycki[6] discusses glass-forming ability from both kinetic and structural standpoints and concludes that the only practical way to determine whether or not a substance will form a glass is by experiment.

Fused silica is a valuable optical material because of its broadband transmission from 180 μm in the ultraviolet to 4.5 μm in the infrared. But the flame hydrolysis technique by which this material is manufactured leaves some water in the finished silica and this water causes optical absorption near 3.0 μm (see Figure III-11). If pure fused silica can be produced in the clean environment of space, a more pure product than is possible on earth might result. Such a pure fused silica could reasonably be expected to show improved optical transmission in both the ultraviolet and infrared.

The oxides of aluminum, zirconium, and yttrium have not been successfully produced in the glassy state on earth. These materials have a low viscosity when molten and devitrify readily when cooled. In the clean environment of space, possibly the absence of contamination that can serve as nuclei for crystal growth would

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(3) W. H. Zachariasen, J. Am. Chem. Soc. 54, 3841-3851 (1932).

(4) K. H. Sun, J. Am. Ceram, Soc. 30, 277-281 (1947).

(5) A. Winter, J. Am. Ceram. Soc. 40, 54-58 (1957).

(6) J. Zarzycki, Glasses - Their Salient Chemical and Physical Properties, In Chemical and Mechanical Behavior of Inorganic Materials, ed. A. W. Searey et al, Wiley Interscience (1970).

permit undercooling and, therefore, glassy alumina, zirconia, and yttria. It is not possible to predict with any accuracy the properties of a material that has never been produced, but optical transmission in the infrared beyond 5.0 μm seems like a realistic possibility.

If glassy alumina, zirconia, and yttria prove impossible to produce in space, there is a possibility that early experiments would show that it is possible to produce polycrystalline materials of more closely controlled crystallite size than is possible on earth. If this proves to be the case, then it would be well to follow with processes designed to produce spinels, which are interesting from an optical standpoint since they are isotropic. Properties of these materials are given in Figure III-12.

Yttria is produced in a transparent polycrystalline state under the trade name of Yttralox (General Electric Company). Both alumina and zirconia are usually polycrystalline and translucent to opaque although small glassy spherules of both have been produced experimentally[7].

All three have melting points above 2000°C and, providing they can be made to remain in the glassy state, make glasses capable of withstanding sustained temperatures of at least 1000°C. Chemical behavior of all three in the crystalline state is adequate and should be so in the glassy state.

Single oxides are proposed for initial processing to avoid problems from phase separation or selective volatilization that might arise in a mixed-oxide composition.

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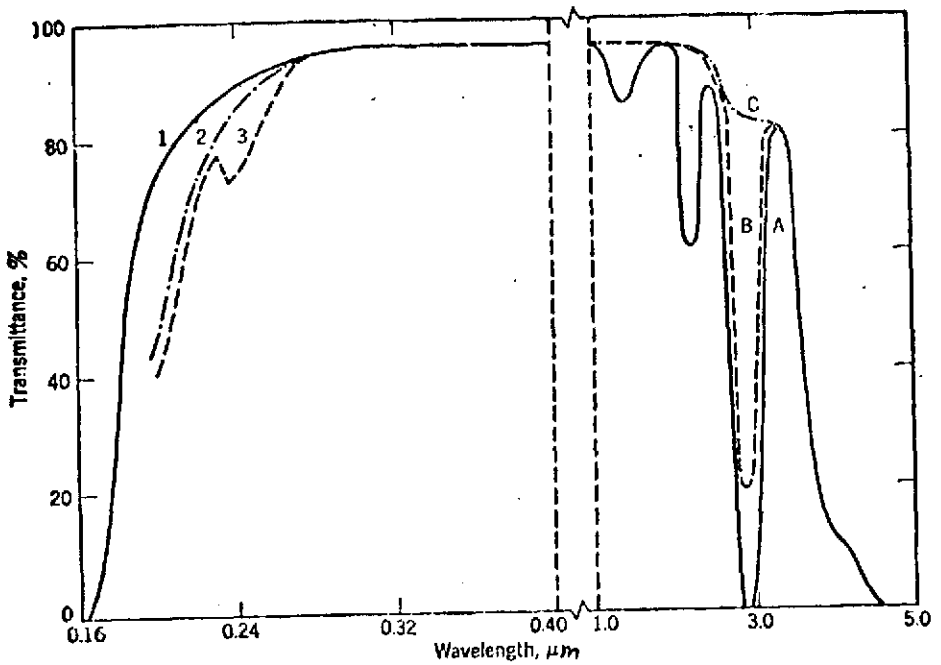
(7) Final Report, Study of the Production of Unique New Glasses, SD 72-SA-0083, Contract NAS8-28014, Space Division, North American Rockwell Corporation.



A - PREPARED BY VAPOR-PHASE HYDROLYSIS.

B - APPROXIMATE REPRESENTATION OF FUSED QUARTZ (GENERAL ELECTRIC, AMERSIL). MINIMA AT 2.73 M MAY VARY ± 20% FROM THAT SHOWN.

C - MELTED IN DRY ATMOSPHERE.

Figure III-11. Transmission Curves for Vitreous Silicas, Thickness 1 CM

MATERIAL	DENSITY (g/cm^3)	MELT PT (°C)	LINEAR THERMAL EXPANSION (MULTIPLY BY 10^{-7}/°C)	INDEX OF REFRACTION
Al 0	4.0	2070	79-119	1.73 (?)
Y 0	4.5	2410	81-94	1.92 2.13-2.20
ZrO	5.2	2690	72-144	--

Figure III-12. Properties of Candidate Materials

III.2.1 Processing Approach

The goal of this approach is to cool the candidate oxides from the melt in sections large enough to be useful for lenses, prisms, and windows without crystallizing. Zero "G" provides an opportunity for containerless melting which will avoid contaminating the surface of the melt with nucleation sites. It is, however, not certain that surface nucleation is the only source of crystal growth. If nucleation occurs internally, then there will be no gain from containerless melting and more research will be required to determine the complete mechanism of crystallization and means to avoid crystal formation.

Assuming that one or all of these oxides can be cooled to a rigid amorphous state, the next problem will be to determine limitations on cooling rates and how they are affected by sample size. As the molten spheres cool they will transfer heat from their interior by radiative transfer and temperature differences within the spheres will be small. But as the oxide continues to cool, the predominant wavelengths are in the long infrared region, to which the material is opaque. As the material hardens and radiative transfer is diminished, temperature difference within the sample will increase and thermally induced stress will rise.

How rapidly this stress rises and what cooling rate is required to keep this stress within acceptable limits are questions that will have to be determined. Once thermal conductivity and diffusivity, thermal expansion, specific heat, and density of the glass are determined; it will be possible to calculate the required cooling rate. Cooling rates and the degree of annealing that they produce are dependent on specimen size and it may well turn out that for each oxide there is a limiting specimen size, beyond which the cooling rate becomes impractically slow.

This limiting specimen size may be the determining factor in deciding whether to eventually produce quantities of product in discrete sections or in continuous rods or bars. The cross section of any specimen must be large enough that the desired lens, prism, or window to be finished from it can be cut or ground directly from the specimen. Re-melting on earth would only return the material to the crystalline state.

Since the physical properties of the proposed amorphous oxides are not known, it is not possible to calculate in advance whether or not this cooling problem will prove to be an obstacle or a minor consideration.

Since optical quality is required in the end product, homogeneity will undoubtedly prove to be an appreciable problem. Preparation of the preformed blanks that will be melted in space will require the most sophisticated techniques possible. These preforms must be free of all impurities, solid or gaseous. Solid impurities will provide nucleation sites and gaseous impurities will remain in the melt as blisters or seeds. Samples of the required purity may require development of ceramic forming techniques better than any now existing.

Design of forming equipment for the first samples to prove that the candidate oxides can be produced in the amorphous state should not present a major problem. A furnace and a specimen holder capable of separating the molten specimen from the root stock and a levitation system should prove to be applications of existing technology.

Appendix B in Book 2 of Volume II of this report discusses the technology involved in Transparent Oxide Processing in Space. The generic process for providing the transparent oxides is given in Figure III-13. Alternative process steps are listed below:

III.2.1.1 Obtaining the Candidate Materials

III.2.1.1.1 Initial Oxide Materials

a. Silica - The possibility of reacting silicon and oxygen under rigorously controlled, water-free, contaminant-free conditions might be considered.

b. Alumina

c. Zirconia

d. Yttria

e. Mixtures - These would be considered for advanced phases of effort.

III.2.1.1.2 Material Purity

a. High Purity

b. Commercial Grades - Since Space Processing is a possible approach to purification, the lower cost of commercial grade materials may benefit the economics of commercial production.

III.2.1.2 Loading the Processing System

a. On the Ground

b. In Orbit - Manual versus automatic.

In general, space operations are more constrained by manpower availability, time, etc. On the other hand, the flight loads and design problems for immobilizing and sequencing specimens may require costly solutions.

III.2.1.3 Melting

a. Thermal Imaging - Including solar, carbon arc, vapor arcs

b. Laser - CO_2

c. Induction - Via tungsten susceptor.

d. Microwave - Not practical for dielectrics.

e. Electron Beam - Needs good vacuum.

Temperature levels, dwell times, rates of heating (and cooling) exert key influences on purity and structure of product. Typical temperature/time plots considered for space processing of the oxides are given in Figures III-14 and III-15.

III.2.1.4 Position Control

a. Acoustic - Potentially undesirably high sound pressures.

b. Electromagnetic - Very high frequencies required for poor conductors.

c. Electrostatic - Requires active control.

d. Gas Stream - Low power, independent of material.

III.2.1.5 Forming

a. Spin - Rotation of melt during cooling can produce spheroidal shapes.

b. Centrifugal Casting - Difficult to prevent devitrification.

c. Electrostatic - Not likely for these materials.

d. Sheet Plate - Requires physical forming, possible devitrification.

III.2.1.6 Cooling (See Figures III-14 and III-15)

a. Radiation - Free

b. Cold Gas - Inert, highly possible.

c. Cryogenic - Work on small samples.

d. Annealing - Problem with devitrification.

III.2.1.7 Eject and Collect from Heating/Cooling Equipment

These functions will likely utilize same process approach as positioning (D. above).

III.2.1.8 Transport, Finish, Utilize

The returned boules will require the same cutting, polishing, cleaning as ground-based products.

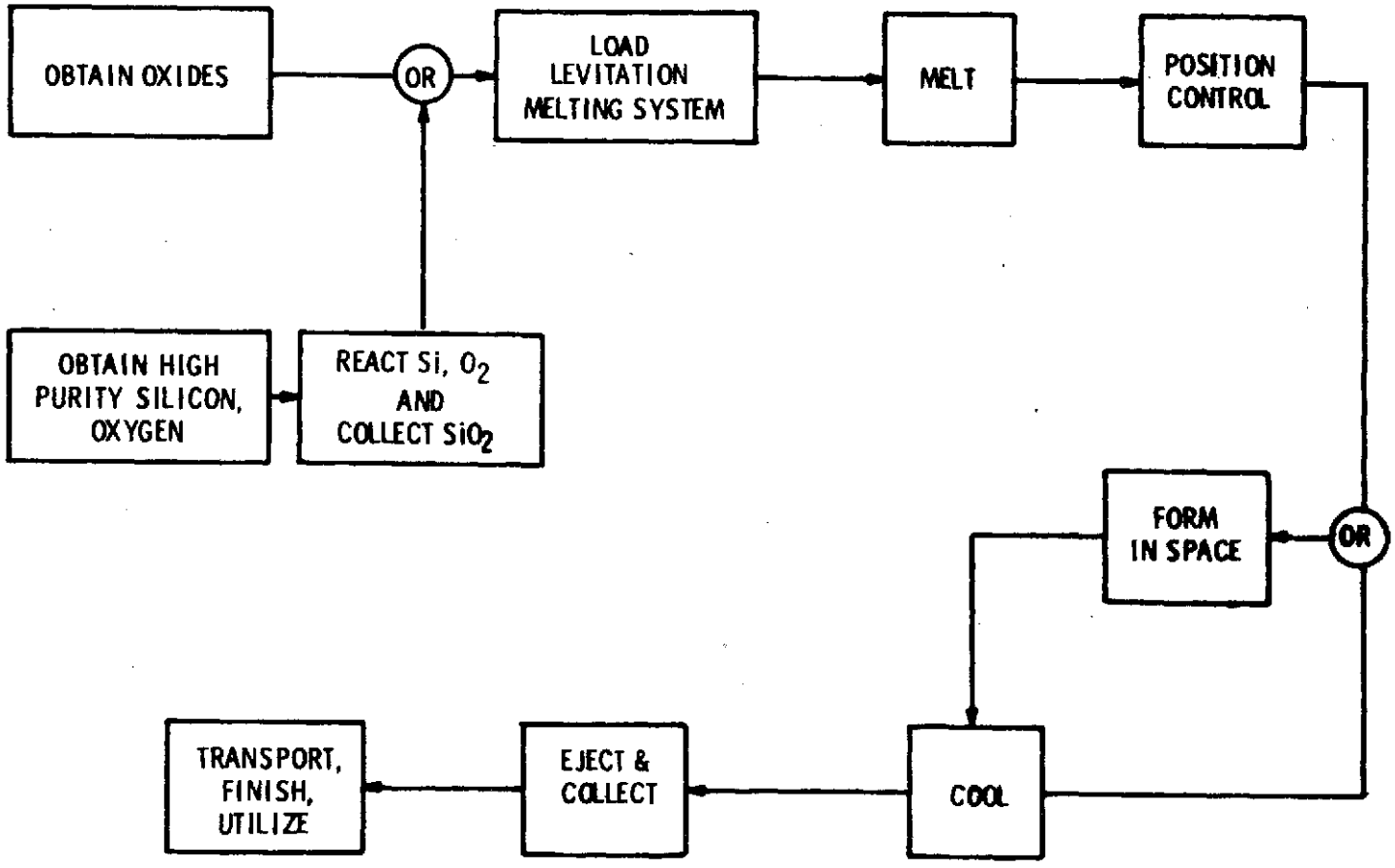


Figure III-13. Transparent Oxide Processing

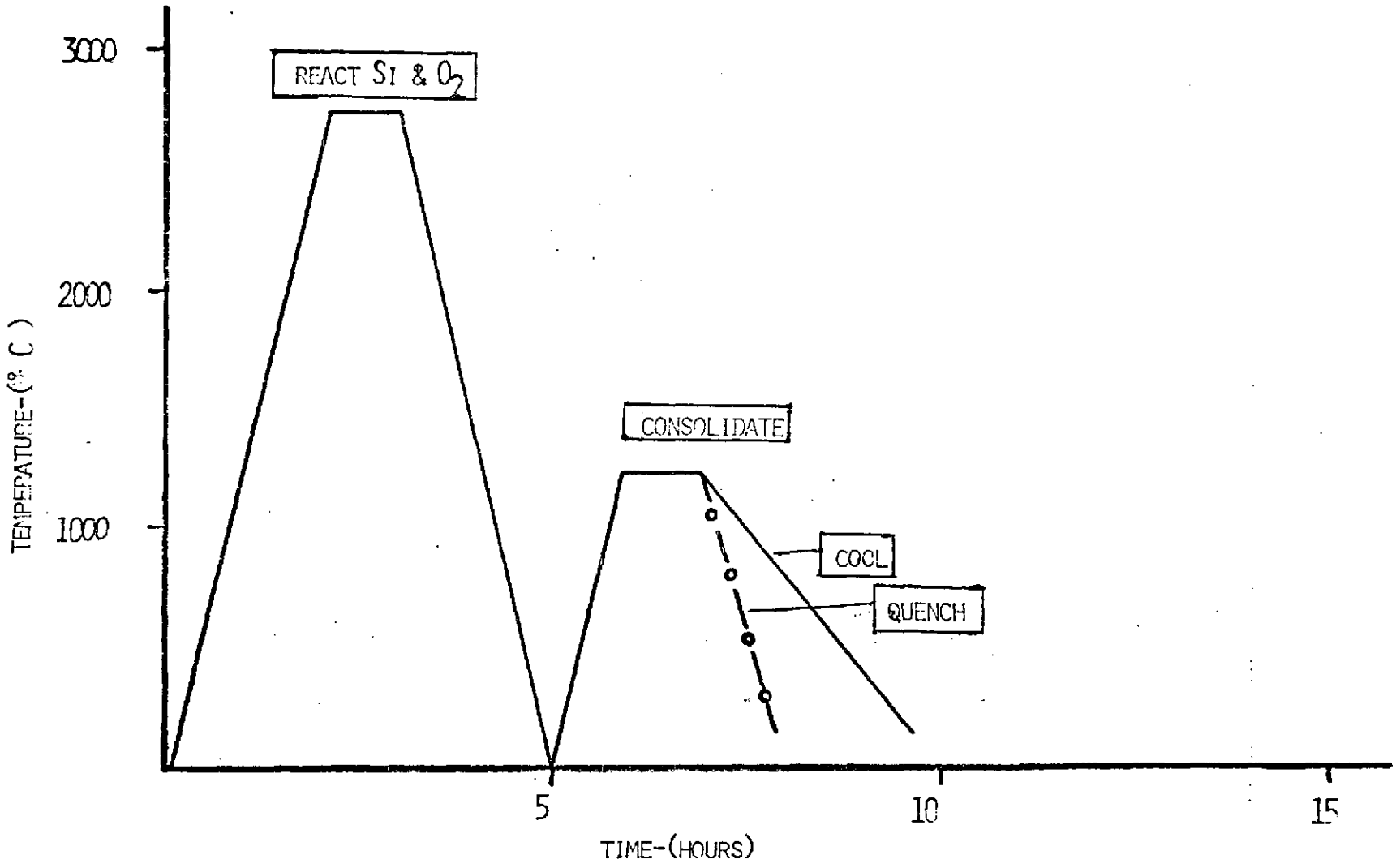


Figure III-14. Potential Heating/Cooling Cycle for Vitreous Silica

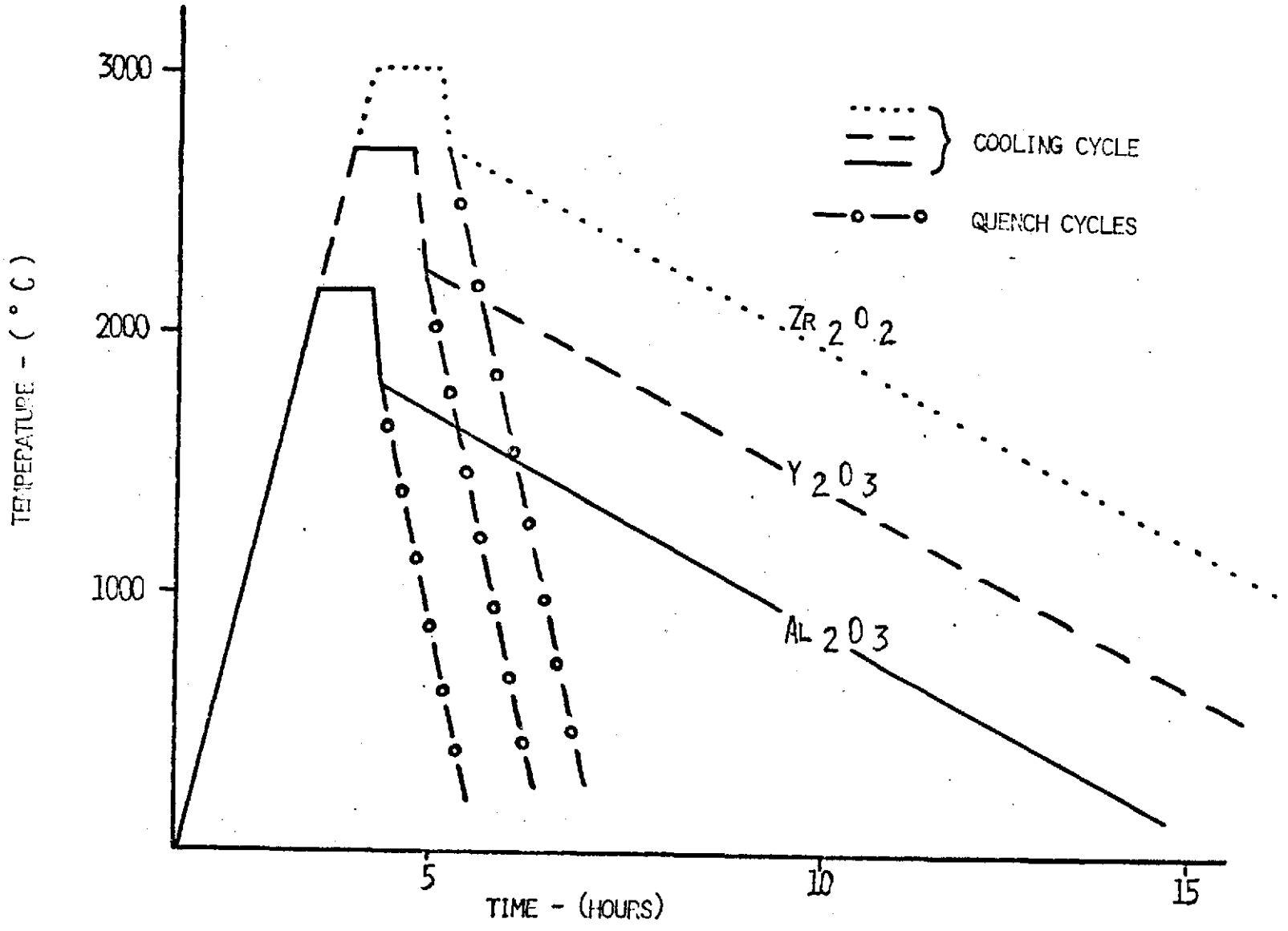


Figure III-15. Potential Heating/Cooling Cycle for Alumina, Zirconia, Yttria

III.2.2 COMPARISON OF APPROACHES FOR PROCESSING TRANSPARENT OXIDES

The broad scope of alternatives listed in the preceding paragraphs was narrowed down to a manageable number through consultations and analyses, such as that given in Appendix B, by experts in processing and materials. Subsequently, a detailed comparison was made of key alternatives against a set of criteria aimed at providing insight into the technological and business aspects of each alternative. Utilizing the scientific and engineering background of the User and the Space Division Study Team, and projecting anticipated technological advances, judgement of each alternative was performed, based on the criteria listed below in their relative order of importance.

1. Relative quality of expected results.
2. Relationship of needs for products.
3. Relative cost of identified approach.
4. Process utilization potential to other products or services.
5. Relative size and weight of space borne equipment.
6. Relative magnitude and variety of support utilities and ground support services.
7. Relative size and weight of materials to be processed.
8. Complexity of process functions.
9. Complexity of support operations.
10. Degree of automation feasible in order to perform process function.
11. Level and tolerance of required environmental conditions.
12. Duration and precision of timing.
13. Precision of performance.
14. Relative magnitude of wastes.

Figure III-16 presents the matrix of alternatives and criteria, with the value judgements compiled from the User/Study evaluation.

	LEVEL & TOLERANCES OF REQUIRED ENVIRONMENT (1)	REQUIRED PRECISION OF PERFORMANCE (1)	DURATION OF PERFORMANCE AND PRECISION OF TIMING (1)	COMPLEXITY OF FUNCTIONS (2)	COMPLEXITY OF SUPPORT OPERATIONS (2)	DEGREE OF AUTOMATION FEASIBLE (2)	RELATIVE SIZE, WEIGHT OF SPACE-BORNE EQUIPMENT (3)	RELATIVE MAGNITUDE, VARIETIES OF SUPPORT UTILITIES, GROUND SUPPORT SERVICES (3)	RELATIVE MAGNITUDE OF RAW MATERIALS (3)	RELATIVE MAGNITUDE OF WASTES (1)	IMPACT ON RELATIONSHIP OF NEED TO AVAILABILITY (4)	RELATIVE IMPACT ON QUALITY OF RESULTS (5)	RELATIVE COST IMPACT (3)	POTENTIAL FOR OTHER USES (3)	WEIGHTED TOTAL	SELECTION RATIONALE
PROCESS STEP OBTAIN OXIDES																
SILICA	5/5	4/4	–	–	5/10	–	5/15	5/15	5/15	4/4	5/20	–	5/15	–	103	← GOOD PAYOFF HERE BUT
ALUMINA	5/5	5/5	–	–	5/10	–	5/15	5/15	5/5	4/4	4/16	–	5/15	–	100	← POTENTIAL OF
ZIRCONIA	5/5	5/5	–	–	5/10	–	5/15	5/15	5/15	4/4	4/16	–	5/15	–	100	← OTHER OXIDES FOR
YTTRIA	5/5	5/5	–	–	5/10	–	5/15	5/15	5/15	4/4	4/16	–	5/15	–	100	← BREAKTHROUGH
MIXTURES	5/5	5/5	–	–	4/8	–	5/15	5/15	5/15	4/4	4/16	–	5/15	–	98	WARRANTS FURTHER WORK.
REACT Si, O_2 ON GROUND	2/2	3/3	–	–1/–2	5/10	–	5/15	3/9	5/15	5/5	3/12	–	3/9	–	78	A PROCESS GOAL IS PURIFICATION. CLOSENESS OF RESULTS SHOWS NEED OF FURTHER INFORMATION.
REACT Si, O_2 IN ORBIT	3/3	2/2	–	–3/–6	1/2	1/2	2/6	2/6	3/9	4/4	2/8	–	1/3	–	39	
HIGH PURITY	5/5	4/4	–	–	4/8	–	5/15	4/12	5/15	5/5	–	–	4/12	–	76	
COMMERCIAL GRADE	5/5	5/5	–	–	5/10	–	4/12	5/15	4/12	4/4	–	–	5/15	–	78	← NEED OF FURTHER INFORMATION.
LOAD LEVITATION MEASURING SYSTEM																
ON GROUND	3/3	5/5	5/5	5/10	3/6	–	4/12	3/9	–	–	5/20	–	5/15	–	85	
IN-ORBIT, AUTOMATIC	5/5	4/4	4/4	2/4	4/8	–	3/9	4/12	–	–	4/16	–	2/6	5/15	83	RELATIVELY SIMPLE, SHORT,
IN-ORBIT, MANUAL	5/5	4/4	3/3	3/6	5/10	–	5/15	5/15	–	–	5/20	–	3/9	–	87	← NON PRECISION TASK
MELT																
LASER	3/3	2/2	–	3/6	4/8	4/8	4/12	2/6	–	–	5/20	–	3/9	5/15	89	HIGH FAMILIARITY
ELECTRON BEAM	2/2	1/3	–	2/4	3/6	4/8	3/9	2/6	–	–	5/20	–	2/6	5/15	79	SOLAR FURNACE
MICROWAVE	4/4	4/4	–	3/6	5/10	5/10	5/15	3/9	–	–	5/20	–	4/12	5/15	105	← WORTH PURSUING
SOLAR FURNACE	4/4	4/4	–	4/8	3/6	4/8	3/9	5/15	–	–	4/16	–	5/15	5/15	100	AS ALTERNATIVE
HIGH RATE	5/5	5/5	5/5	5/10	5/10	5/10	4/12	4/12	–	–	–	5/25	4/12	5/15	121	← CLOSE RESULTS
MODERATE RATE	3/3	5/5	4/4	5/10	5/10	5/10	5/15	5/15	–	–	–	4/20	5/15	5/15	122	INDICATES WORK NEEDED
SUPERHEAT	4/4	–	4/4	–	–	–	4/12	–	–	–	–	5/25	4/12	5/15	72	← TESTING NEEDED
MELT TEMPERATURE	5/5	–	5/5	–	–	–	5/15	–	–	–	–	4/20	5/15	4/12	72	TO RESOLVE THIS
POSITIONING AND EJECTION																
ELECTROSTATIC	5/5	5/5	4/4	4/8	4/8	5/10	5/15	5/15	–	5/5	–	5/25	4/12	5/15	127	← SUBSTANTIALLY
ACOUSTIC	5/5	5/5	3/3	3/6	4/8	5/10	4/12	4/12	–	5/5	–	5/25	3/9	5/15	115	BETTER
RF	5/5	4/4	3/3	4/8	5/10	5/10	3/9	3/9	–	5/5	–	5/25	2/6	5/15	109	
GAS JET	4/4	5/5	5/5	5/10	3/6	5/10	2/6	3/9	–	3/3	–	4/20	5/15	5/15	108	
FORMING																
SPIN	4/4	4/4	3/3	4/8	3/6	4/8	4/12	4/12	–	–	3/12	4/20	4/12	4/12	113	
CENTRIFUGAL CASTING	2/2	2/2	2/2	2/4	2/4	2/4	1/3	3/9	–	–	4/16	2/10	2/6	5/15	77	
ELECTROSTATIC	3/3	3/3	1/1	1/2	1/2	2/4	3/9	2/6	–	–	5/20	4/20	2/6	4/12	88	FINISHING OPERATIONS REQUIRED ON
SHEET-PLATE PRESS	1/1	1/1	4/4	3/6	4/8	3/6	2/6	3/9	–	–	3/12	5/25	3/9	2/6	78	
NONE	5/5	5/5	5/5	5/10	5/10	5/10	5/15	5/15	–	–	3/12	5/25	5/15	–	127	← GROUND IN ANY CASE
COOLING																
RADIATION	4/4	4/4	3/3	3/6	5/10	4/8	5/15	5/15	–	5/5	–	3/15	5/15	5/15	106	
COLD GAS QUENCH	5/5	5/5	4/4	5/10	4/8	5/10	4/12	3/9	–	3/3	–	4/20	4/12	5/15	113	← SUPERCOOLING
CRYO QUENCH	5/5	5/5	5/5	4/8	3/6	5/10	1/3	2/6	–	2/2	–	5/25	4/12	5/15	102	GENERALLY USED IN GROUND
ANNEAL	3/3	3/3	2/2	2/4	1/2	3/6	3/9	2/6	–	5/5	–	2/10	4/12	4/12	74	LABS, MAY NOT BE REQUIRED IN SPACE, MORE DATA NEEDED.

Figure III-16. Comparison Of Alternatives For Space Processing Of Transparent Oxides

III.2.3 DEFINITION OF SELECTED APPROACH TO PROCESSING TRANSPARENT OXIDES

Figure III-17 summarizes all the alternatives discussed in previous paragraphs and indicates what appears, at this time, to be the most feasible processing approach for transparent oxides. In addition, the figure indicates those areas in which no clear decision could be made.

FOLDOUT FRAME

FOLDOUT FRAME

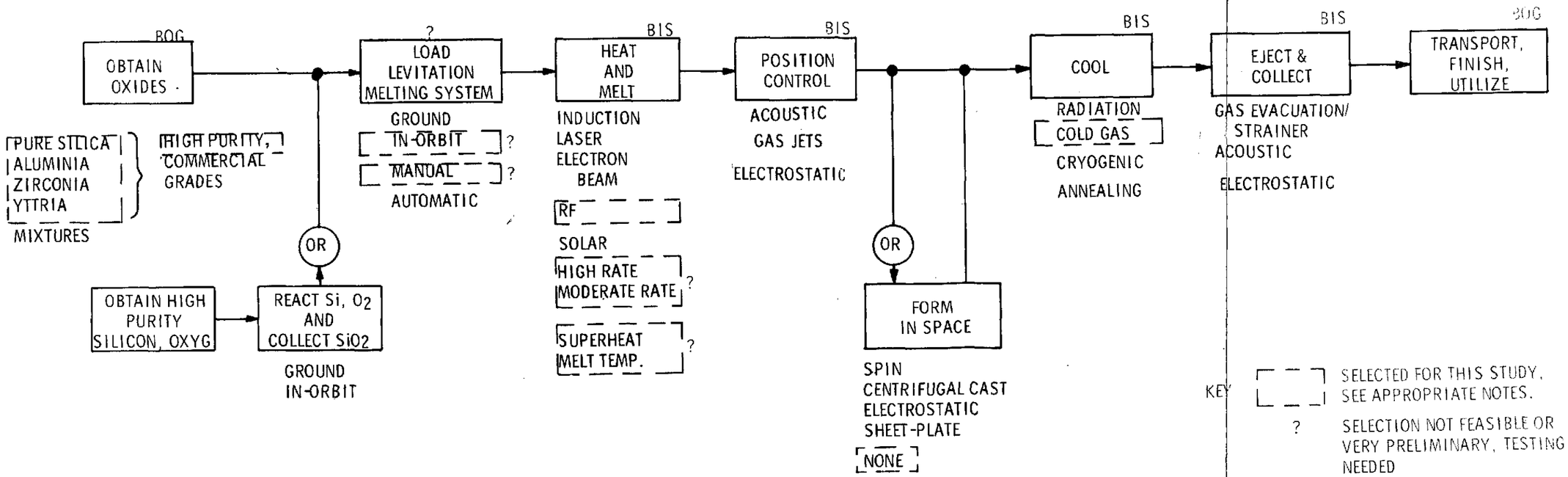


Figure III-17. Definition Of Best Implementation Approach For Transparent Oxide Processing

III.3 SELECTION OF BEST APPROACH FOR PROCESSING OF HIGH PURITY TUNGSTEN X-RAY TARGETS

The x-ray producing surface of an x-ray tube target must have a high atomic number for x-ray production and a high melting point to withstand up to 10^7 watts per square centimeter power input levels from the electron beam which impinges on the target to create x-rays. The problem is, perhaps, best expressed in the statement of Figure III-18. At the present time, pure tungsten or a tungsten alloy is the most logical choice. The target must have good high temperature strength, but, more importantly, it must have ductility at and above room temperature. Therefore, a very desired property of the target material would be a pure tungsten with a ductile brittle transition temperature (DBTT) below room temperature.

> AN X-RAY TUBE IS A GLASS ENCAPSULATED ELECTRON-BEAM FURNACE THAT IS BARELY FRUSTRATED IN ITS EFFORT TO MELT THE CHARGE.

Figure III-18. The X-Ray Target Problem

X-ray tubes using presently available target materials are warranted for 20,000 exposures and a goal is to increase this exposure warranty by a minimum of 50%.

In present practice, a standard life test is used to simulate the wide variety of techniques used by doctors. It is performed as follows: 20,000 exposures at 150 KVP and 250 mA with 1.25 second exposures taken at the rate of two exposures per minute on a 2 mm focal spot. The desired life would be 30,000 or more such exposures with less than a 10% x-radiation fall-off after the 30,000 exposures.

An additional requisite or goal would be a yield strength of 20,000 psi minimum at 1100°C to resist warpage of the target angle.

The increase in customer-required power levels (attainable with the higher amperage generators which became available about five years ago) has made the commercially made solid disc tungsten target unusable at the present time. The impurity level and ductile brittle transition temperature of these targets were high and these targets could be castastrophically fractured in a very few exposures at power level inputs of about 1×10^5 watts per square centimeters.

The majority of present medical x-ray tubes, therefore, are fabricated from an expensive tungsten/10% rhenium alloy layer which is bonded to a molybdenum substrate. The rhenium now added to the tungsten target surface layer to increase ductility is about $2,200 per KG ($1000 per pound) and accounts for about one-third of the target cost. If a sufficiently ductile tungsten could be developed, it would be much more fabricable and, thus, eliminating rhenium, could be less expensive to produce.

An appreciation for the need for better target materials may be gained by noting the wide variety of target approaches listed in Figure III-19 that have most recently been evaluated by GE in the search for improved performance.

III.3.1 PROCESSING APPROACHES

The problems of producing tungsten with room temperature ductility are discussed in Appendix A, Volume II, Book 2. Laboratory measurements show that the material produced by ground-based powder-metallurgy techniques has a large recrystallized grain size and the yield stress increases sharply as the temperature decreases from $600^{\circ}K$, so that at about $400^{\circ}K$ the yield stress exceeds the fracture stress. The ductility falls sharply with temperature and is essentially zero at $475^{\circ}K$. The brittle fracture stress is essentially independent of temperature below the ductile to brittle transformation temperature (DBTT). Below the DBTT, failure

1. PURE TUNGSTEN (G.E., FANSTEEL, METALLWERK PLANSEE PHILLIPS ELMET)
2. 0.5% RHENIUM/TUNGSTEN COMPOSITES
3. 3% RHENIUM/TUNGSTEN COMPOSITES
4. 5% RHENIUM/TUNGSTEN COMPOSITES
5. 10% RHENIUM/TUNGSTEN COMPOSITES
6. CVD TUNGSTEN ON MOLY AND TUNGSTEN
7. CVD RHENIUM/TUNGSTEN ON MOLY
8. CVD TUNGSTEN ON GRAPHITE
9. PLASMA SPRAYED TUNGSTEN ON GRAPHITE
10. PLASMA SPRAYED TUNGSTEN ON MOLYBDENUM
11. PLASMA SPRAYED TUNGSTEN ON TUNGSTEN
12. ELECTRODEPOSITED TUNGSTEN ON TUNGSTEN
13. ELECTRODEPOSITED TUNGSTEN ON MOLYBDENUM
14. ELECTROPLATED RHENIUM AND IRON ON TUNGSTEN
15. SINGLE CRYSTAL ZONE REFINED TUNGSTEN
16. SINGLE CRYSTAL ZONE REFINED MOLYBDENUM

Figure III-19. Target Design and Materials Most Recently Evaluated

occurs by intercrystalline fracture. This behavior is typical of tungsten produced by powder metallurgy techniques.

Considerable laboratory testing indicates that such failure is not only a function of grain size, but also of interstitial impurity content (mainly carbon and oxygen), and surface features (cracks, absorbed layers).

While impurity content can, with sufficient processing, be controlled in ground processing, grain size, and possibly surface condition, appear to be better controllable via containerless processing.

Two major containerless processes have been considered:

- o Levitation Melting, and
- o Float Zone Refining

These process steps are pictured in Figure III-20.

The alternative process approaches, and alternatives for each process step, are discussed below.

III.3.1.1 Levitation Melting

III.3.1.1.1 Selection and Preparation of Material for Processing

As a basis for evaluation of processes, a number of possible tungsten powder and rod material purities have been selected:

a. Tungsten powder of greater than .999 purity; metallic impurity content (eg., Mo, Si, K, Ca, etc.) of approximately 800 ppm total and the interstitials (C, O_2, N_2, H_2) having a total impurity content of 100 ppm. For example: C, 20 - 40 ppm; O_2, 20 - 40 ppm; N_2 and H_2, 2 - 0 ppm.

b. Tungsten rod or wire the same as (a) above.

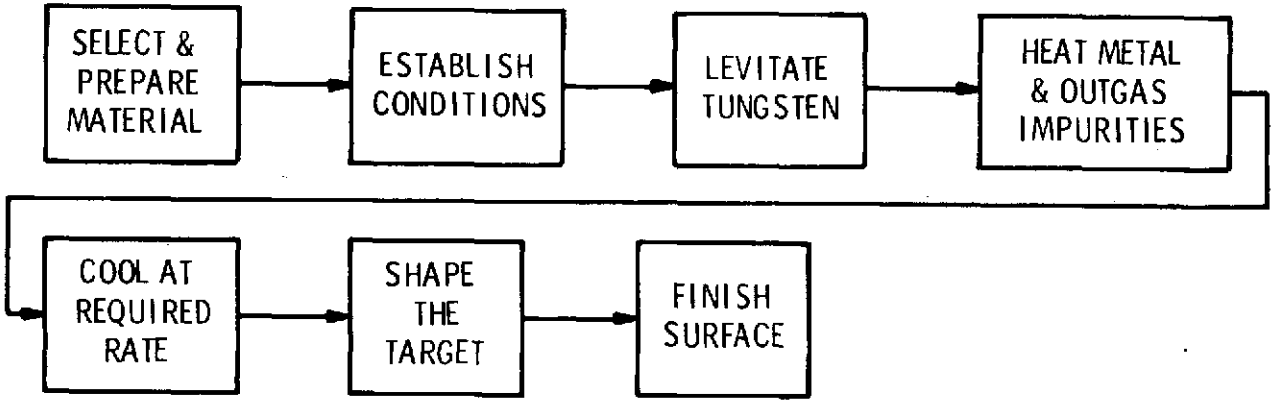
c. Tungsten rod or wire of .999 purity with the metallics totaling about 80 ppm and the interstitials a total of 20 ppm.

d. Tungsten float-zoned rod of .99999 with metallic impurities and interstitials at 10 - 20 ppm.

e. The quantity of material required to shape one target from a levitated sphere is .6 KG (1.45 pounds) for a .0056 M (1/4 inch) thick solid tungsten target. With possible scrap losses, .8 KG (1.75 pounds) is a reasonable quantity.

f. In normal commercial target manufacture by forging, the excess machined from the outer diameter accounts for about .227 KG (1/2 pound). Therefore, a minimum quantity of .91 KG (2 pounds) per target would be required.

(A) LEVITATION MELTING



(B) FLOAT ZONE REFINING

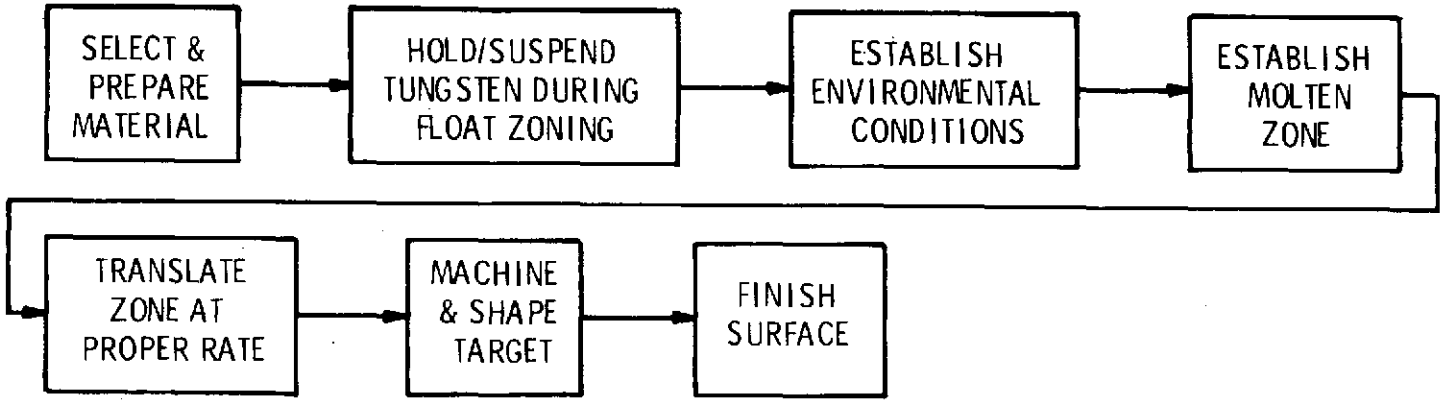


Figure III-20. High Purity Tungsten X-Ray Target Processing

g. The present ring-type target provides a .0011 M (0.005 inch) thick ring surface layer which constitutes the focal track or x-ray producing surface of the target. For a .0675 M (3 inch) diameter target, a thin ring of this type could be joined to an appropriate target and this ring would require .096 KG (0.21 pound) of tungsten.

h. A small .014 M (5/8 inch) diameter of .014 M (5/8 inch) square by .00135 M (0.06 inch) thick sample to be joined to a standard tungsten/10% rhenium target in the focal track region. Weight of .0072 KG (0.016 pounds).

III.3.1.1.2 Levitation and Positioning of the Tungsten During Processing

a. Radiofrequency positioning levitation at zero gravity.

b. Ground-based levitation and positioning at one gravity.

c. Radiofrequency positioning, solar heating, zero gravity.

d. Radiofrequency positioning, electron beam heating, zero gravity.

III.3.1.1.3 Environmental Conditions for Melting

a. High vacuum -- 10^{-3} N/M^2.

III.3.1.1.4 Melting Temperature Profile

a. Slow rise time (1000°C per hour).

b. Fast rise time (1000°C per minute).

c. Dwell at temperature < melt.

d. No dwell at temperature < melt.

e. Momentary dwell at temperature > melt (1 to 5 minutes).

f. Prolonged dwell at temperature > melt (30 minutes to 1 hour).

g. No dwell at temperature > melt.

h. Slow cooling rate -- (10 - 100°C per hour).

i. Fast cooling rate -- (100 - 1000°C per minute).

j. Fast cooling rate plus quench.

III.3.1.1.5 Shaping of the target

a. The target may be shaped by spinning during melting and solidification

b. A spherically shaped levitation ball may be formed by cold mechanical working under controlled environmental conditions.

c. A spherically shaped levitation-melted ball may be formed by hot mechanical working under controlled conditions.

d. The levitation melted sphere (or shape of any type) may be cast into a cooled metal mold (water-cooled copper is used).

e. Target may be formed by machining.

III.3.1.1.6 Surface cleaning

a. Chemical cleaning - electropolishing.

b. Mechanical.

c. None.

III.3.1.2 Float Zone Refining

III.3.1.2.1 Select and Prepare Materials for Processing

The same alternatives as III.3.1.1.1 except all samples (powders) would first be formed into cylindrical rods.

III.3.1.2.2 Float Zone Technique

a. The float zone rod is normally held at each end while the heating coil moves along the rod diffusing impurities in front of the "high heat" zone.

b. In zero gravity, the ideal suspension method would be to suspend the rod at each end by RF coil levitation forces.

III.3.1.2.3 Environmental Conditions of Zone Refining

The same alternatives as written under III.3.1.1.3.

III.3.1.2.4 Zone Refining Temperature Profile

The RF coil must be moved along the axis of the tungsten cylinder so that the high temperature float zone provides high diffusion rates for the impurities to continuously diffuse toward one end of the cylinder.

a. Multiple passes are normally made to increase the purity of the material on each pass.

b. The translation of the RF coil along the axis of the rod is normally governed by impurity diffusion. Thus slow translation of the float zone at .0113 M - .09 M per hour (0.3 inch - 4 inches per hour) would be required.

c. Slow cooling of melted zone.

d. Fast cooling of heated zone -- preferred.

e. RF heating -- logical choice.

III.3.1.2.5 Shaping of the Target

Any diameter zone-refined could be sliced into sections and then:

a. Shaped by cold working.

b. Shaped by hot working.

c. Completely machined to target size.

III.3.1.2.6 Cleaning

a. Chemically.

b. Mechanically.

c. None.

In order to select the best approach from the large variety of alternatives listed above, scientific and engineering judgement has been employed to reduce the permutations of processes and process steps to those which are sufficiently practical to warrant further consideration. These are shown in Figures III-21A and III-21B.

ALTERNATIVE STEPS	1	2	3	4	5	6	7	8	9	10
A. LEVITATION MELTING										
1. MATERIAL SELECTION PREPARATION										
A. W POWDER (0.999 P., 800 PPM INTERSTITIALS)	X									
B. W ROD (0.999 P., 800 PPM INTERSTITIALS)		X	X	X	X		X	X		
C. W ROD OR WIRE 0.9999 PURITY						X				
D. W ROD FLOAT ZONE REFINED (0.99999)										
2. QUANTITY OF REFINED TUNGSTEN/TARGET										
A. 0.91 KG (BASED ON SOLID W TARGET)	X				X			X		
B. 0.096 KG (BASED ON 77 MM DIA. RING), 15 MM SPHERE		X	X	X		X	X			
3. METHOD OF HEATING AND LEVITATION										
A. RF HEAT & POSITIONING, ZERO-G LEVITATION	X	X		X	X	X				
B. RF POSITIONING, HEATING & LEVITATION										
C. RF POSITIONING, SOLAR HEATING, ZERO G LEVITATION			X					X		
D. RF POSITIONING, ELECTRON BEAM HEATING, ZERO-G LEVITATION							X			
4. HEATING TEMP. PROFILE										
4.1.A SLOW TEMP. RISE										
4.1.B FAST TEMP. RISE	X	X	X	X	X	X	X	X		
4.2.A DWELL AT TEMP. BELOW MELTING (E.G., 2400°C TO ATTAIN PRELIMINARY DEGASSING)	X	X	X	X	X	X	X	X		
4.2.B NO DWELL AT TEMP. BELOW MELTING										
4.3.A MOMENTARY DWELL AT TEMP ≥ MELT.			X							
4.3.B PROLONGED DWELL AT TEMP ≥ MELT.	X	X	X	X	X	X	X	X		
4.3.C NO DWELL AT HIGHEST TEMP.										
4.4.A RAPID COOLING (EG., 100-1000°C/MIN.)	X	X	X		X	X	X	X		
4.4.B SLOW COOLING (E.G., 100-1000°C/HR.)										
4.4.C RAPID COOLING PLUS QUENCHING AFTER SOLIDIFICATION				X						
5. ENVIRONMENTAL CONDITIONS DURING PROCESS										
A. HIGH VACUUM	X	X	X	X	X	X	X	X		
B. LOW PARTIAL PRESSURE OF INERT GAS										
6. SHAPING OF THE TARGET										
A. SPINNING DURING LEVITATION MELTING								X		
B. FORMING (AT CONTROLLED ENVIRONMENT) FORM THE LEVITATION-PRODUCED SPHEROID	X	X	X	X		X	X			
C. MACHINING					X					
7. CLEANING OF SURFACE										
A. CHEMICAL PROCESS (E.G., ELECTRO-POLISHING)	X	X	X	X		X				
B. MECHANICAL PROCESS										
C. NONE NECESSARY										

Figure III-21A. High Purity Tungsten X-Ray Targets Alternative Processes

	1	2	3	4	5	6	7	8	9	10
B. FLOAT ZONE REFINING										
1. SELECT AND PREPARE MATERIAL INTO RODS										
A. FROM W POWDERS (0.999 PURITY, 800 PPM INT.)									X	
B. FROM W MATERIAL 0.9999 PURITY										X
C. FROM FLOAT ZONE W WIRE (0.99999 PURITY)										
2. METHOD OF HOLDING/SUSPENDING DURING FLOAT ZONE REFINING PROCESS										
A. ATTACHMENT OF ROD AT BOTH ENDS IN ONE-G ENVIRONMENT										X
B. ATTACHMENT OF ROD AT BOTH ENDS IN ZERO-G ENVIRONMENT									X	
C. RF-POSITIONING OF ZERO-G LEVITATED ROD										
3. NUMBER OF FLOAT ZONE PASSES										
A. SINGLE PASS									X	
B. MULTIPLE PASSES										X
4. ZONE REFINING TEMPERATURE PROFILE										
4.1.A TRANSLATION RATE SLOWER THAN MAX. (GOVERNED BY IMPURITY DIFFUSION)									X	X
4.1.B TRANSLATION RATE = MAX. (AS GOVERNED BY IMPURITY DIFFUSION)										
4.2.A FAST COOLING OF ZONE									X	X
4.2.B SLOW COOLING OF ZONE										
5. ENVIRONMENTAL CONDITIONS										
A. HIGH VACUUM									X	X
B. INERT GAS										
6. SHAPING OF TARGET AFTER SLICING ROD										
A. COLD WORKING									X	
B. HOT WORKING										X
C. MACHINING										
7. CLEANING OF SURFACE										
A. CHEMICALLY									X	
B. MECHANICALLY										X
C. NONE NECESSARY										

Figure III-21B. High Purity Tungsten X-Ray Targets Alternative Processes

III.3.2 COMPARISON OF APPROACHES FOR PROCESSING TUNGSTEN X-RAY TARGETS

The ten combinations of process steps for the two processes shown in Figures III-21A and III-21B were next evaluated by the User/Space Division Study Team utilizing consultation from metallurgical and process engineering personnel, as required. Judgement of each combination was based on a standard set of criteria listed below in their relative order of importance.

1. Level and tolerance of required environmental conditions.
2. Relative quality of expected results -- high purity and small grain structure (i.e., best removal of impurities).
3. Duration and precision of time -- control of temperature profile and rapid cooling control.
4. Degree of automation feasible in order to perform process function.
5. Relative cost of identified approach.
6. Relative size and weight of space borne equipment and material to be processed.
7. Complexity of process functions.
8. Complexity of support operations.
9. Relative magnitude and variety of support utilities and ground support services.
10. Process utilization potential to other products or services.
11. Relationship of needs for products.

Figure III-22 presents the matrix resulting from that judgement, and indicates the combination of process steps that form the selected best approach

III.3.3 DEFINITION OF SELECTED APPROACH TO PROCESSING OF TUNGSTEN X-RAY TARGETS

Figure III-23 summarizes the main features of the approach selected through the above comparison. The simplified diagram given in Figure III-20 for the Levitation Melting Processing of Tungsten X-Ray Targets, when expanded to include all reasonable alternatives, becomes that pictured in Figure III-24.

SELECTION CRITERIA / ALTERNATIVES	RATING WEIGHTED/UNWEIGHTED (1–10)										WEIGHTING FACTOR
	1	2	3	4	5	6	7	8	9	10	
1. RELATIVE QUALITY OF EXPECTED RESULTS--HIGH PURITY AND SMALL GRAIN STRUCTURE (I.E., BEST REMOVAL OF IMPURITIES)	10.5/7	12/8	12/8	12/8	12/8	12/8	12/8	3.0/2	7.5/5	7.5/5	1.5
2. RELATIVE COST OF IDENTIFIED APPROACH	4.2/3	7.0/5	7.0/5	5.6/4	5.6/4	2.8/2	5.6/4	7.2/6	7/5	7/5	1.4
3. DURATION AND PRECISION OF TIME--CONTROL OF TEMPERATURE PROFILE AND RAPID COOLING CONTROL	3.9/3	6.5/5	6.5/5	2.6/2	6.5/5	6.5/5	6.5/5	6.5/5	7.8/6	7.8/6	1.3
4. LEVEL AND TOLERANCE OF REQUIRED ENVIRONMENTAL CONDITIONS	6.0/5	8.4/7	8.4/7	7.2/6	8.4/7	8.4/7	8.4/7	8.4/7	6.0/5	10.8/9	1.2
5. COMPLEXITY OF PROCESS FUNCTIONS	7.2/6	9.6/8	8.4/7	7.2/6	8.4/7	7.6/8	7.2/6	9.6/8	8.4/7	7	1.2
6. COMPLEXITY OF SUPPORT OPERATIONS	6.6/6	8.8/8	6.6/6	7.7/7	7.7/7	7.7/7	6.6/6	9.9/9	5.5/5	7.7/7	1.1
7. RELATIVE SIZE AND WEIGHT OF SPACE BORNE EQUIPMENT AND MATERIAL TO BE PROCESSED	5.5/5	7.7/7	8.8/8	6.6/6	7.7/7	7.7/7	6.6/6	8.8/8	6.6/6	6.6/6	1.1
8. DEGREE OF AUTOMATION FEASIBLE IN ORDER TO PERFORM PROCESS FUNCTION--	7	8	6	8	8	8	8	9	7	9	1.0
9. PROCESS UTILIZATION POTENTIAL TO OTHER PRODUCTS OR SERVICES	7.2/9	5.6/7	5.6/7	5.6/7	5.6/7	5.6/7	5.6/7	5.6/7	3.2/4	3.2/4	0.8
10. RELATIVE MAGNITUDE AND VARIETY OF SUPPORT UTILITIES AND GROUND SUPPORT SERVICES	4.0/5	7.2/9	7.2/9	7.2/9	7.2/9	7.2/9	7.2/9	7.2/9	5.6/7	7.2/9	0.8
11. RELATIONSHIP OF NEEDS FOR PRODUCTS	6.3/9	4.9/7	4.9/7	4.9/7	4.9/7	4.9/7	4.9/7	4.9/7	4.9/7	4.9/7	0.7
WEIGHTED TOTALS	68.4	85.7	80.7	74.6	82	78.4	78.6	80.1	69.5	78.7	

SELECTED

Figure III-22. Approach Selection Matrix For Processing Of Tungsten X-Ray Targets

MATERIAL SELECTION	TUNGSTEN ROD, 0.999 PURE, 800 PPM INTERSTITIALS
TARGET TYPE	77mm DIA RING (15mm DIAM SPHERE, 0.096 KG)
HEATING, POSITIONING	RF, ZERO G LEVITATION
HEATING PROFILE	RAPID RISE
TEMP. DWELL, BELOW MELT	YES
PROLONGED TEMP. DWELL ABOVE MELT	YES
COOLING PROFILE	RAPID
VACUUM OR INERT GAS	VACUUM
SHAPING	GROUND FORGING IN CONTROLLED ENVIRONMENT
SURFACE CLEANING	CHEMICAL

Figure III-23. Selected Approach For Processing Of Tungsten X-Ray Targets

FOLDOUT FRAME

FOLDOUT FRAME 2

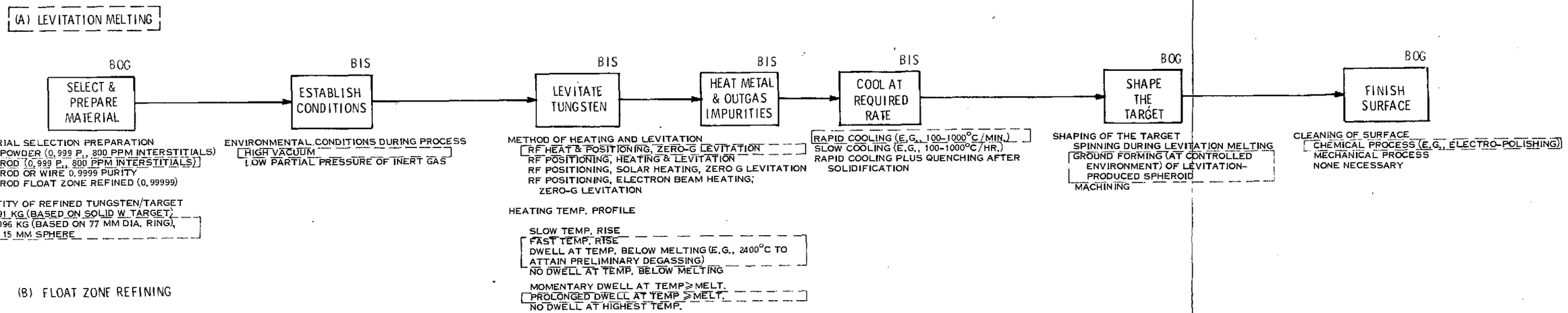


Figure III-24. Definition Of Best Implementation Approach For High Purity Tungsten X-Ray Target Processing

That figure also identifies the approach selected for this Study, and the location (ground or space) at which each process step is judged to be performed best.

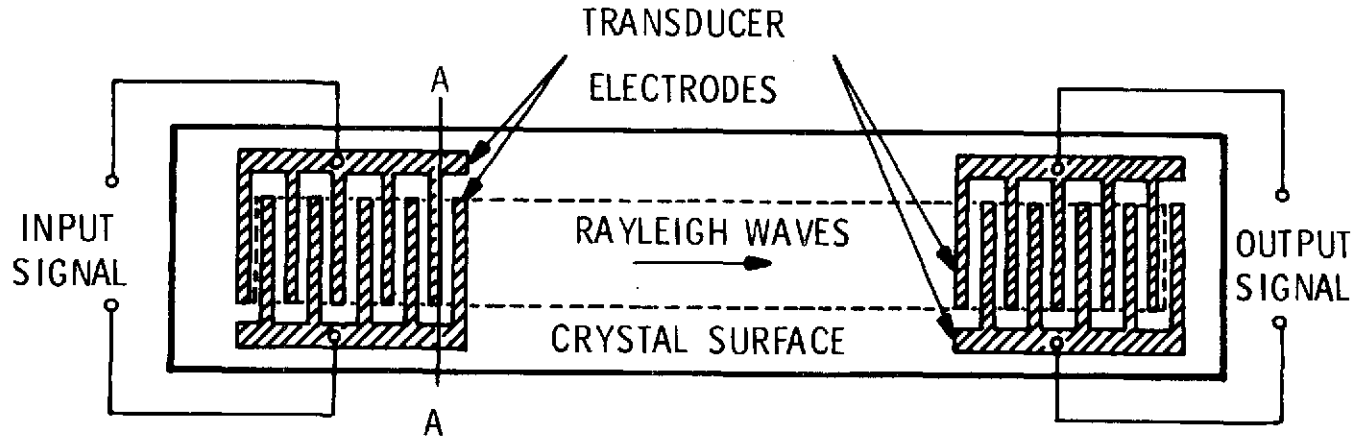
III.4 SELECTION OF BEST APPROACH FOR FABRICATION OF SURFACE ACOUSTIC WAVE COMPONENTS

Surface acoustic wave (SAW) devices have been in use for some time in signal processing functions such as crystal resonators in oscillators and bandpass filters, and as delay lines in volatile memories and circulating integrators.

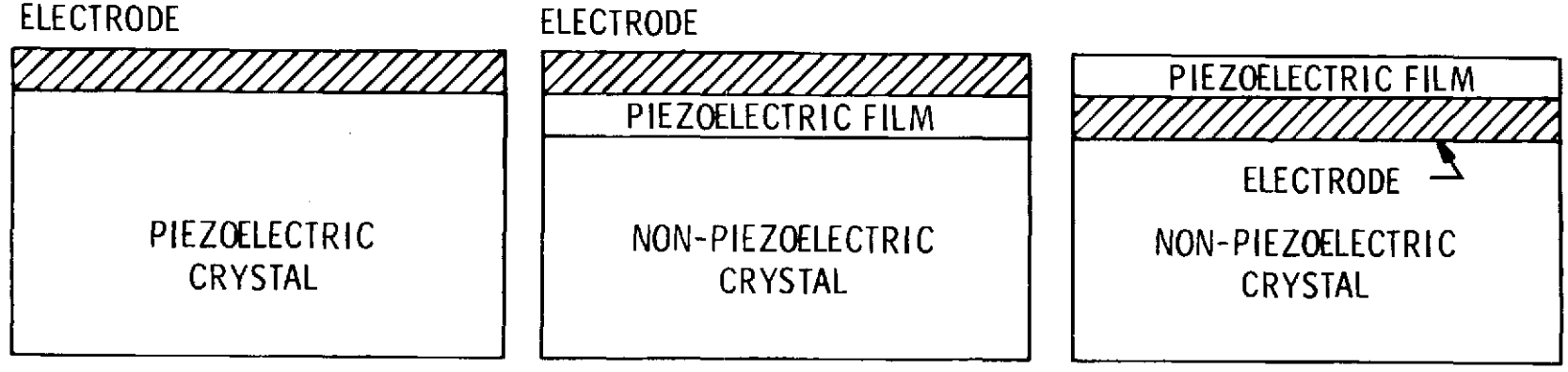
For about the past 10 years, there has been intensive development of more complex signal processing elements. In addition to providing order-of-magnitude improvements in performance, surface acoustic wave devices have been shown to be capable of performing entire system functions. Their advantages include:

- o High frequencies and large bandwidths
- o Easily formed complex circuitry
- o Compatibility with integrated circuit manufacturing techniques
- o Precise reproducibility and design predictability

A typical device, Figure III-25, consists of a piezoelectric input transducer, an elastic body serving as a mechanical resonator or a storage delay medium, and one or more piezoelectric output transducers appropriately placed on the elastic body. Electric impulses into the input transducer electrodes on the crystal surfaces generate acoustic waves through the crystal itself. Since the impulses now move at acoustic velocity (instead of electromagnetic velocity of the original electronic impulses), the effective time delay provides signal compression and temporary storage until the acoustic wave reaches the output transducer where the crystal provides electrical signal outputs. Compression and storage of signals due to the acoustic signal slow velocity are the key



a) WORKING SURFACE



b) TYPICAL ALTERNATIVE STRUCTURES OF SURFACE ACOUSTIC WAVE COMPONENTS
(SECTION A-A)

Figure III-25. Fundamental Surface Acoustic Wave Component

phenomena of this technology. The signal storage and compression enable, with proper patterns of transducer electrodes, the performance of such functions as signal amplification, filtering, etc.

While such components have been built and operated at lower frequencies (below about 4 GHz), there is a growing need for equipment to operate in the millimeter wave range - above about 10 GHz to possibly 30 GHz. Desired usage in radar systems is a driving requirement now. Furthermore, the FCC's formal allocation of bands in that microwave range for private and public use, coupled with NASA's successful 1970 ATS-V demonstration of millimeter wave communications, will open up a tremendous market for such devices. This will be due, primarily to the gains in bandwidth and an order of magnitude increase in available frequencies. At present, advanced SAW device research - for example, the RAC plasma etching at Lincoln Laboratories - for extremely high performance pulse compression signal processors - anticipates a time-bandwidth product hopefully as great as 5000. Bandwidths of 30 GHz, a bandwidth of 9 GHz; with a time-bandwidth product of 5000, the corresponding delay change will be $5000/9 \times 10^9 = 0.6$ μsec. At a velocity of 3000 m/sec, a wave will travel 1.8 mm in this time.

Designing conservatively, we can therefore visualize the typical 30 GHz SAW device:

- o Maximum length: $\sim$ 2mm
- o Finger spacing (and width): 250Å
- o Number of fingers: $1.8 \times 10^{-3}/500 \times 10^{-10} = 36{,}000$
- o Length of fingers: 0.1 mm

Half of the electroded area will be covered by fingers, and half will be bare. With 36,000 fingers, each 250Å wide, and an equal number of gaps, the length,

overall, of the complete 30 GHz SAW device will be less than two millimeters. It appears, therefore, that crystal size is not a problem for such high frequency components, but that quality of the crystal - especially its surface over the two millimeter lengths - will be the primary difficulty.

High velocity substrates could increase the finger spacing, as well as the SAW crystal size. Use of diamond as a substrate would increase sizes almost three times; perfect 6 mm diamonds are not yet economically attractive, however. Sapphire is an excellent non-piezoelectric crystal substrate offering a 2:1 size increase, which is not excessively expensive. However, deposition of a piezoelectric film is required; 1 GHz sapphire devices have been demonstrated, using deposited Aluminum Nitride piezoelectric films. Their performance is not yet well known for higher frequency, high performance devices.

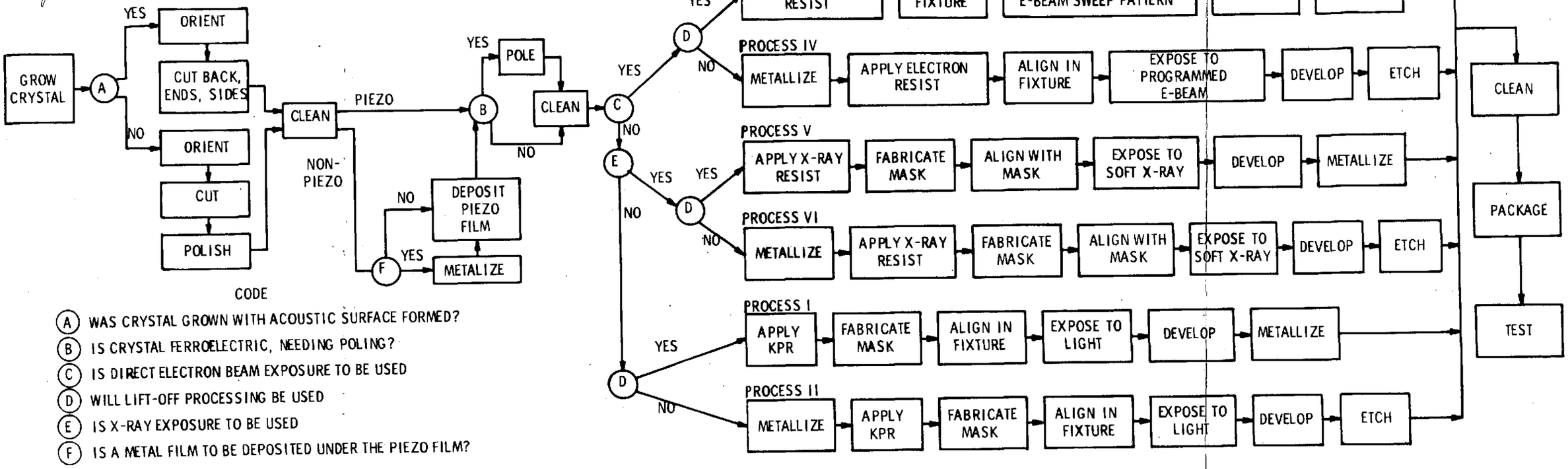
From the foregoing analysis, it is clear that high frequency operation will require millimeter size crystals. Imperfections should always be less than wavelength size, and hence essentially non-existent for 30 GHz devices. Conversely, 10 GHz devices will require larger crystals ($\sim$6 mm) with much less stringent quality requirements.

III.4.1 PROCESSING APPROACHES

Figure III.26 summarizes the major process steps and options involved in the fabrication of surface acoustic wave components. The basic process, options and alternatives are discussed below.

There are, then, two major problems facing the fabrication of the required 10-30 GHz componentry. In order of difficulty, they are: (1) The imprinting of the extremely fine circuitry on the crystal, now beyond the state-of-the-

FOLDOUT FRAME 1



CODE

(A) WAS CRYSTAL GROWN WITH ACOUSTIC SURFACE FORMED?
(B) IS CRYSTAL FERROELECTRIC, NEEDING POLING?
(C) IS DIRECT ELECTRON BEAM EXPOSURE TO BE USED
(D) WILL LIFT-OFF PROCESSING BE USED
(E) IS X-RAY EXPOSURE TO BE USED
(F) IS A METAL FILM TO BE DEPOSITED UNDER THE PIEZO FILM?

FOLDOUT FRAME 2

Figure III-26. Surface Acoustic Wave Device Processing

art for the best printing or photographic processes on earth; and (2) The other is the availability of crystals with the required degree of perfection.

III.4.1.1 CRYSTALS

Piezoelectric crystals that are most suitable for surface acoustic wave applications are, basically, those that have a high electromechanical coupling, low loss and low temperature coefficient; also they should be available in large sizes and at reasonable costs.

Since, in general, it is unlikely that any individual material would excel in all of these characteristics, the most suitable materials are selected after considering the tradeoffs involved.

Following the choice of material, the crystal class must be determined. For surface wave generation, propagation and detection, the elastic, piezoelectric and dielectric properties are used to associate the optimum directions for pure mode propagation, high electromechanical coupling and lower temperature coefficient, which can be combined into an optimum choice of the direction of surface wave propagation in a crystal wafer.

The inherent physical properties of the crystal significantly affect the final operating performance of the working device. These properties are of two types; (1) the actual composition and stoichiometry of the material, and (2) the physical properties of the surface finish.

For optimum operation, the structure of the crystal must be completely homogeneous and free of inclusions, strains and defective growth patterns. Small changes in crystalline direction will cause a very slight change in the acoustic surface wave propagation velocity, thus causing severe phasing problems, especially in such devices as tapped or dispersive delay lines.

The second important property is the surface propagating condition. High frequency operation of acoustic surface wave devices requires that the substrate be optically polished, flat to $\lambda/20$ and stress free. Any scratches or stresses may cause the surface waves to become dispersive and, in severe cases, could cause a prohibitive attenuation loss of the propagating wave. Surface layer conditions can cause changes in surface wave velocity as small as 0.001 per cent which, in turn, may cause phase mismatches that are totally unacceptable in some devices.

Most earth-grown materials used for surface wave propagation are Czochralski- or flux-grown and frequently suffer from imperfections and inhomogeneities. One possible way to improve the quality of crystals may be to grow them in an orbiting environment.

Zero gravity, and, possibly, vacuum are the two main properties that could be useful for growing crystals in space. Although several methods, currently in use, such as high pressure hydrothermal, vapor phase and solution growth, could produce certain of the desirable crystals, the most likely choice of growth process is either an adpatation of the Czochralski or flux growth method. Epitaxial growth from the vapor may be utilized for deposition of a piezo layer if a non-piezo substrate is required for a specific application. Growth of extremely high quality crystals could be beneficial to a host of applications, aside from surface wave devices. Further discussion of surface acoustic wave crystals is given in Appendix C, Book 2, Volume II.

Major alternatives in obtaining crystals are as follows.

III.4.1.1.1 Materials

a. Piezoelectric

- Lithium Niobate
- Quartz
- Bismuth Germanate
- Zinc Oxide
- Aluminum Nitride
- Cadmium Sulphide
- Ferroelectric Ceramics
- Gallium Arsenide

b. Non-Piezoelectric

- Sapphire
- Rutile
- Silicon

Since the non-piezoelectric materials cannot provide the transducing function, they require a thin surface coating of piezoelectric crystal. A typical combination is Aluminum Nitride/Sapphire.

III.4.1.1.2 Crystal Growth Method

- Float Zone
- Czochralski Pulling
- Solution Growth Epitaxial Vapor Growth
- High Pressure Hydrothermal
- Vernuil Process

III.4.1.1.3 Growth Environment

- Ground
- Space

All of the crystal growing methods listed are ground-based, and development efforts will be required to adapt them to operation in the orbital environment, if space-growth of crystals appears desirable.

III.4.1.1.4 Crystal Orientation, Cutting, Polishing

Before cutting crystals into substrate wafers, it is necessary to utilize x-ray diffraction equipment to establish the required crystal planes which possesses

the proper coupling coefficients, etc. If it becomes possible to promote growth of a crystal with a surface having the desired plane, then it may be possible to simplify this step. Thus alternatives are:

a. Alignment; cutting, polishing all six surfaces

b. Alignment; cutting, polishing back, sides, ends only

c. Performed on Ground

d. Performed in Space

While there appear to be no technical advantages to performing this process in space, if the crystal is grown in space, and is later to be imprinted in space, there may be some economic or administrative advantage in minimizing the transportation requirements.

III.4.1.1.5 Crystal Cleaning

The effects of surface contamination on the performance of surface acoustic wave components are so potentially catastrophic that "cleaning" appears prior to many process steps.

a. Chemical and ultrasonic baths are standard. Further, ultra-cleaning may be by:

- Back sputtering;

- Ion beam scrubbing, and again, as noted earlier, there are the alternatives of cleaning in the environment of;

- Ground,

- Space

III.4.1.2 Coatings

Various coatings are required in the alternatives which were considered for fabricating surface acoustic wave components.

III.4.1.2.1 Surface Metallization

Deposition of a metallic (usually gold) surface is required in several process steps. Growth of such a layer, in a preferential pattern, on non-piezoelectric crystal wafers encourages the subsequent growth of the piezoelectric layer, and results in higher coupling, higher quality components and better performance. The alternatives are:

a. Sputtering

b. Vapor Deposition

c. Ground-based

d. Space-based

III.4.1.2.2 Application of Resist Coating

The resist coating provides the specific surface treatment which subsequently enables the reaction of the selected energy source to form the desired electrode pattern. The specific coating is a function of the energy source to be used in the exposure process step. Alternative coatings are:

a. Electron Resist

b. X-Ray Resist

c. Photo Resist, with application in;

d. Ground-based, or

e. Space-based Facilities.

III.4.1.3 Imprinting the Electrode Patterns

In order to appreciate the difficulties anticipated in working towards the goal of components capable of operating in the 10-30 GHz frequency range, it is useful to calculate sizes involved in the formation of electrode "finger" patterns on the surface of crystals forming the high frequency surface wave devices. An average value for the speed of propagation of surface waves on a

suitable crystal material is about c = 3000 m/sec. At a frequency f = 30 GHz, the wavelength of an acoustic surface wave is then, correspondingly,

$$\lambda = c/f \qquad \frac{3000}{30 \times 10^9} = 0.1 \times 10^{-6} \text{ meter}$$

A "normal" surface pattern for interdigital electrodes in cross section is shown in Figure III-25. With equal electrode "finger" and "gap" widths, fingers normally sized at $\lambda/4$ width must be conducting lines .025 microns = 250Å wide. The visible spectrum extends down to a wavelength of about 0.4 microns, and hence is not suited to form patterns with this fine spacing, or for the lithographic reproduction of such patterns.

The difficulties of meeting the high frequency goals are, therefore, those of forming such fine lines, as well as of providing crystal surfaces perfect enough in small detail to permit the formation of electrode patterns with such fine resolvable fingers. Optical resolution is not likely to be sufficient. Ultraviolet radiation extends down to wavelengths of about 0.4Å, but no suitable processing techniques are available for utilizing that energy. The x-ray spectrum extends roughly from 100Å (soft) to less than .01Å (hard). Soft x-ray resist techniques exist, and so it should be possible to develop a lithographic method for meeting the 250Å finger width requirements by x-ray reproduction from a mask pattern, providing means for making the mask is found.

Certainly, formation of mask patterns this fine is not feasible by the usual method of photographic reduction, even with diffraction limited lenses. The only contender for mask pattern formation in this resolution range at present is electron beam exposure. Electron beam sizes of the order of 100Å are feasible. However, energy density over the beam cross section tends to be Gaussian, so it

is difficult to predict whether a nominal 100Å beam will produce a sufficient defined edge for a line only 250Å wide.

Surface acoustic wave electronic components currently being produced in ground facilities are limited, because of interaction of the terrestrial environment (specifically seismic vibration) with the equipment utilized in imprinting the circuitry, to operating at frequencies less than 4 GHz. Attempts have been made to solve the problem by eliminating these very low frequency vibrations from the imprinting systems utilized in earth processing. Even with 200-ton concrete base plates, isolation at very low frequencies has not been effective. For electrodes of 1 micron width, an accuracy of ± 0.1 micron should be maintained. For a vibration frequency of 1 Hz, with peak value of 10^{-4}G, the peak-to-peak amplitude of motion would be of the order of 50 microns, far larger than could be tolerated. In addition, the amplitude increases by approximately an order of magnitude for each 0.3 Hz decrease in vibration frequency.

It is felt that isolation from such vibration may be achievable in a properly designed spacecraft facility, thus perhaps enabling the use of an electron beam gun in the imprinting process to make it possible to produce the fine circuitry for frequencies as high as, possibly, 30 GHz. A discussion of low frequency vibration in spacecraft is given in Appendix D, Book 2 of Volume II.

Key process steps, and their alternatives, involved in the imprinting phase of fabricating surface acoustic wave components are discussed below,

III.4.1.3.1 Mask Fabrication

The mask is essentially the "stencil" pattern for certain alternatives for forming required surface acoustic wave component circuitry. Since fabrication

of the mask does not involve the crystal, this is not a sequential step in the process. This is one of the most critical steps in several of the alternatives, since the mask must be even more accurate than the circuitry it is to produce. Alternatives, based on the exposure energies considered are:

a. Photolithographic Mask

b. X-Ray Lithographic Mask

c. Ground-based

d. Space-based

III.4.2 COMPARISON OF APPROACHES FOR FABRICATING SURFACE ACOUSTIC WAVE COMPONENTS

III.4.2.1 Evaluation of Crystal Substrates

The major parameters sought for in potential materials for the surface acoustic wave component crystals are listed in Figure III-27, together with the rationale for their choice.

PARAMETER	ADVANTAGES
(1) HIGH VELOCITY OF PROPAGATION	HIGH FREQUENCY OPERATION; DESIRABLE FOR CLOSELY SPACED TAPS IN A TAP DELAY LINE
(2) MODERATE TO HIGH COUPLING COEFFICIENT	YIELDS WIDE BANDWIDTHS AND LOW INSERTION LOSS
(3) EASILY GROWN OR DEPOSITED HIGH QUALITY CRYSTALS	FIDELITY OF HIGHLY PURE CRYSTALS
(4) LARGE LENGTHS	LONG DELAY TIMES
(5) LOW COST	IMPLEMENTATION INTO MANY SYSTEMS (LARGE QUANTITIES). CAN BE DISCARDED IF DAMAGED IN PROCESSING (YIELDS LOW COST)
(6) CAN BE CLEAVED OR EASILY POLISHED	YIELDS LOW COST FOR CRYSTAL SURFACE PREPARATION

Figure III-27. Desired Characteristics of a Surface Wave Substrate

Applying those parameters as criteria on the specific materials felt to be most competitive at this time results in the comparison pictured in Figure III-28.

Of the piezoelectrics, Lithium Niobate has the best figure of merit. It can be stated that this material is the prime choice for many surface wave device applications. That does not necessarily define it as the ultimate choice, but indicates that it should be the principal consideration when one is selecting a substrate for a surface wave device. Since, however, specific applications impose specific requirements for substrate properties, there will be instances where it will have to be ruled out.

The choice for non-piezoelectric materials is sapphire. It is especially desirable since it is available in long lengths and is compatible with piezoelectric film (ZnO, CdS, AlN) growth on its surface. Although Silicon can be grown in fairly large sizes, sapphire has the advantage of an extremely low acoustic attenuation. In addition, silicon films can be grown on sapphire for implementing some of the active devices that are possible using silicon material.

III.4.2.2 Evaluation of Alternative Processing Approaches

Dialogs between the Space Division Study Team and Drs. Tehon and Wanuga, supplemented by consultations with experts in various aspects of the alternative approaches provided the scientific and engineering judgement required to evaluate those alternatives. Figure III-29 summarizes that evaluation, indicates the selected alternative, and provide a brief rationale for that selection. As part of that evaluation process, referring to Figure III-26, many of the coded decisions for a high frequency, high performance lithium niobate SAW device can now be determined.

CRYSTAL	PIEZOELECTRICS						
	HIGH VELOCITY (3)	COUPLING COEFFICIENT (2)	GROWTH PROPERTIES (4)	SIZES (1)	COST (1)*	SURFACE FINISH (5)	WEIGHTED FIGURE OF MERIT
LITHIUM NIOBATE	(3)	(1)	(2)	(2)	(2)	(2)	33
QUARTZ	(3)	(3)	(2)	(2)	(2)	(2)	37
BISMUTH GERMANATE	(4)	(2)	(3)	(3)	(2)	(2)	43
ZINC OXIDE	(3)	(2)	(2)	(4)	(3)	(2)	38
ALUMINUM NITRIDE	(2)	(3)	(4)	(4)	(4)	(3)	51
CADMIUM SULFIDE	(4)	(2)	(4)	(4)	(3)	(3)	54
FERROELECTRIC CERAMICS	(3)	(1)	(4)	(1)	(1)	(4)	49
GALLIUM ARSENIDE	(3)	(4)	(3)	(3)	(3)	(2)	45
			NON PIEZOELECTRICS				
SAPPHIRE	(2)	(5)	(2)	(1)	(2)	(1)	32
RUTILE	(2)	(5)	(2)	(3)	(3)	(2)	40
SILICON	(3)	(5)	(2)	(2)	(2)	(2)	41

*SPACE GROWTH OF CRYSTALS WILL LIKELY ELIMINATE THIS AS A COMPARISON MEASURE

WEIGHTING FACTOR

(1) MOST IMPORTANT

(5) LEAST IMPORTANT

RELATIVE RATING KEY

(1) EXCELLENT
(2) GOOD
(3) MODERATE
(4) POOR
(5) UNACCEPTABLE

Figure III-28. Figure Of Merit For Surface Wave Substrates

FOLDOUT FRAME 1

ALTERNATIVES	RATING	SELECTION/RATIONALE
SUBSTRATE MATERIAL		
PIEZOELECTRIC		
LITHIUM NIOBATE	1	← BEST COMBINATION OF ACOUSTIC, ELECTROACOUSTIC, PHYSICAL, COST PROPERTIES. SAPPHIRE CLOSE CONTENDER.
QUARTZ	3	
BISMUTH GERMANATE	4	
ZINC OXIDE	4	
ALUMINUM NITRIDE	7	
CADMIUM SULFIDE	7	
FERROELECTRIC CERAMICS	3	
GALLIUM ARSENIDE	6	
NON-PIEZOELECTRIC		
SAPPHIRE	2	
RUTILE	5	
SILICON	4	
CRYSTAL GROWTH METHOD		
FLOAT ZONE REFINING	2	
CZOCHRALSKI PULLING	1	← TWO TOP-RATED APPEAR BEST COMBINATION OF GROWTH SPEED, POWER REQUIRED, EQUIPMENT WEIGHT & COMPLEXITY, QUALITY OF PRODUCT. MORE DATA NEEDED.
SOLUTION GROWTH	1	
VAPOR GROWTH "	2	
HIGH PRESSURE HYDROTHERMAL	4	
VERNUIL PROCESS	3	
SPACE	1	← BEST POTENTIAL FOR QUALITY, SIZE, SPEED.
GROUND	2	
CRYSTAL ORIENTATION, CUTTING, POLISHING		
ONE SURFACE IN PRINCIPAL PLANE, CUT & POLISH BACK, SIDES, ENDS ONLY	1	← PREFERRED, IF POSSIBLE. BEST ALIGNMENT. ELIMINATES STEPS.
ALIGN, CUT, POLISH 6 SURFACES	2	
GROUND	1	← NO SPACE ADVANTAGE. HIGH COMPLEXITY IN SPACE.
SPACE	2	

FOLDOUT FRAME 2

ALTERNATIVES	RATING	SELECTION/RATIONALE
CRYSTAL CLEANING		
CHEMICAL BATH) REQUIRED		← PRECLEANING, BEST ON GROUND
ULTRASONIC BATH)		
BACK SPUTTERING	1	
ION BEAM SCRUBBING	1	← ULTRACLEANING PRIOR TO COATING SURFACE REQUIRED.
GROUND	1	BACK SPUTTERING IS CLOSE CONTENDER.
SPACE	1	← PREFERRED
SURFACE METALLIZATION		
SPUTTERING	1	← HIGH PURITY REQUIRED. VAPOR DEPOSITION IS
VAPOR DEPOSITION	1	STRONG CONTENDER, NEED DATA.
GROUND	1	
SPACE	1	← PREFERRED, MINIMIZE SURFACE CONTAMINATION.
APPLICATION OF RESIST COATING		
ELECTRON RESIST	2	
X-RAY RESIST	1	← SEE EXPOSURE
PHOTO RESIST	3	
GROUND	1	
SPACE	1	← PREFERRED
FABRICATION OF MASK		
PHOTOLITHOGRAPHIC MASK	2	
X-RAY LITHOGRAPHIC MASK	1	← SEE EXPOSURE
GROUND	2	
SPACE	1	← BASED ON VIBRATION ISOLATION REQUIRED FOR EXTREME FREQUENCY RESOLUTION
EXPOSURE		
PROGRAMMED ELECTRON BEAM	2	
SOFT X-RAY	1	← HIGHEST RESOLUTION
LIGHT	3	
GROUND	2	
SPACE	1	← BASED ON VIBRATION ISOLATION. CLEANLINESS OF HARD VACUUM IS ADVANTAGE.
ALL OTHER STEPS		
GROUND	1	← NO CRITICAL REQUIREMENTS.

Figure III-29. Comparison Of Alternatives

A Yet determined. Experiments in crystal growing are needed - first, on the ground.

B Yes. Poling of the ferroelectric lithium niobate is required.

C No. Electron beam formation of a mask, for subsequent soft x-ray lithography, is much less time consuming and cheaper.

D No, probably. Lift-off processing preserves the quality of surface, but is difficult. Experiments may be required for final resolution of this process step.

E Yes. This may be the only way to achieve the required electrode finger resolution.

F No (unless aluminum nitride/sapphire experiments prove highly successful).

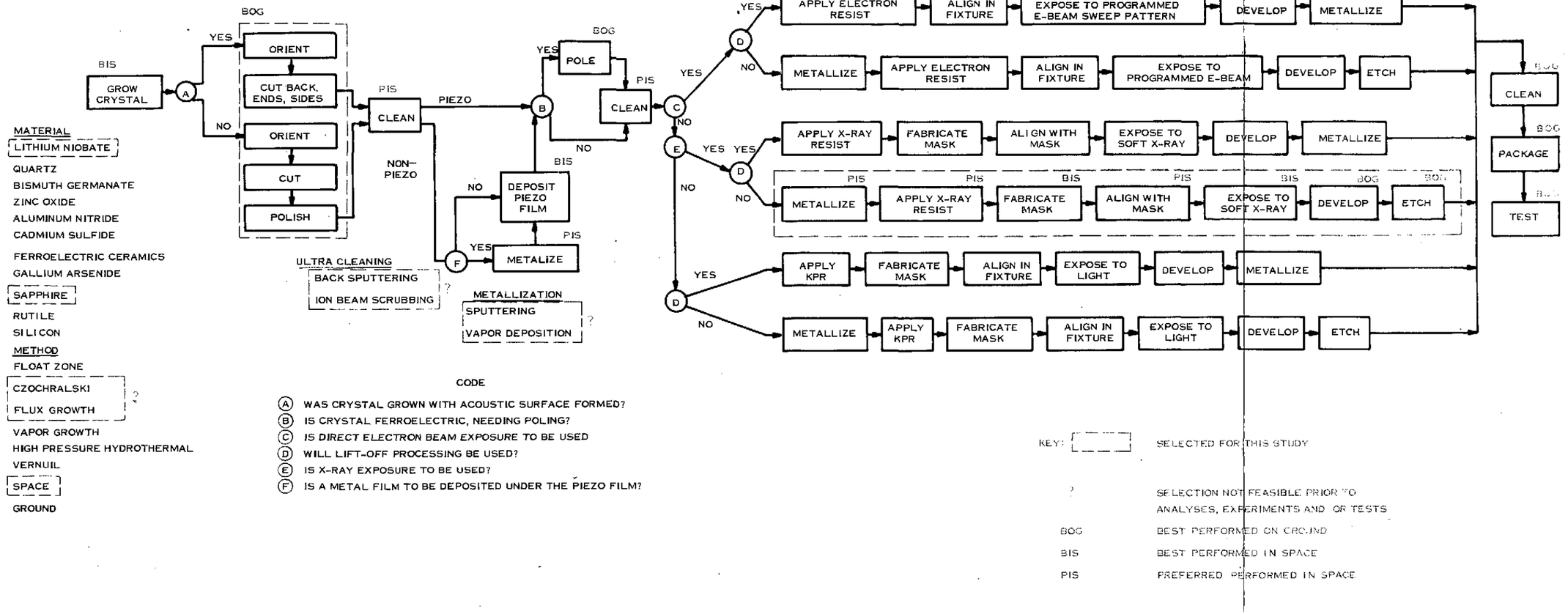
III.4.3 DEFINITION OF BEST APPROACH FOR FABRICATING SURFACE ACOUSTIC WAVE COMPONENTS

Figure III-30 summarizes the overall approach to implementing this concept, lists all the key alternatives, and indicates the alternatives selected at this time. Where present knowledge simply did not allow selections to be made, it is so indicated.

A major feature of this evaluation is the indication that some process steps, while entirely feasible for performance on the ground, might preferably be performed in space for reasons of overall minimization of total process complexity, economy, etc. Such decisions will require later investigation.

FOLDOUT FRAME 1

FOLDOUT FRAME 2



MATERIAL

- LITHIUM NIOBATE
- QUARTZ
- BISMUTH GERMANATE
- ZINC OXIDE
- ALUMINUM NITRIDE
- CADMIUM SULFIDE
- FERROELECTRIC CERAMICS
- GALLIUM ARSENIDE
- SAPPHIRE
- RUTILE
- SILICON

METHOD

- FLOAT ZONE
- CZOCHRALSKI ?
- FLUX GROWTH ?
- VAPOR GROWTH
- HIGH PRESSURE HYDROTHERMAL
- VERNUIL
- SPACE
- GROUND

CODE

(A) WAS CRYSTAL GROWN WITH ACOUSTIC SURFACE FORMED?
(B) IS CRYSTAL FERROELECTRIC, NEEDING POLING?
(C) IS DIRECT ELECTRON BEAM EXPOSURE TO BE USED
(D) WILL LIFT-OFF PROCESSING BE USED?
(E) IS X-RAY EXPOSURE TO BE USED?
(F) IS A METAL FILM TO BE DEPOSITED UNDER THE PIEZO FILM?

KEY: [ ] SELECTED FOR THIS STUDY

? SELECTION NOT FEASIBLE PRIOR TO ANALYSES, EXPERIMENTS AND OR TESTS

BOG BEST PERFORMED ON GROUND

BIS BEST PERFORMED IN SPACE

PIS PREFERRED PERFORMED IN SPACE

Figure III-30. Best Implementation For Fabrication Of Surface Acoustic Wave Components

III.5 DEFINITION OF REQUIREMENTS FOR EXPERIMENTS TO VERIFY SELECTED APPROACH FOR SEPARATION OF ISOENZYMES

III.5.1 CRITICAL ELEMENTS IN SELECTED APPROACH FOR SEPARATION OF ISOENZYMES, AND RELATED KNOWLEDGE "GAPS"

The essential problem to be solved is finding a suitable technique to separate and obtain sufficient quantities (100's of milligrams) of isoenzymes, the chemical process steps to preserve and recover these isoenzymes, and enable their use in the production of anti-bodies. These antibodies are expected to be useful for detection of very small amounts of the specific isoenzymes in patients suspected of specific diseases or damage states.

III.5.1.1 Separation Method

While the state-of-the-art in gel electrophoresis and isoelectric focussing is rapidly growing, the practical limitations of ground operations have inhibited the broad experimental investigation of the effects of variations in controllable process variables. For example, very little information has been obtained for electrophoresis at < 10 volts/cm. The longer separation times and consequent increased convective effects on band broadening are potential problems here. Furthermore, analytical treatment of the problems involved has, in most realistic cases, been shown to be extremely difficult. Ogston[2], for instance, finds "even in these relatively simple cases", which include a continuum of interconnected channels whose sizes are large with respect to the molecules passing through them (he lists electrophoretic migration in a porous medium as an example), "Formidable mathematical difficulties may have to be faced." Furthermore, if one is to account for "Bulk flow (resulting, for example, from electro-endosmosis), then there are also severe problems in hydrodynamics." Such bulk flows also

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[2] Ogston, A.G., On the Physical Chemistry of Porous Systems, British Medical Bulletin, 1966, Volume 22, Number 2.

include convective flows. To add to the analytical difficulties, Ogston also states, "If the porous character of the system is to be useful..., the porous material must itself show some kind of selective interaction with the components of the solution." The gel electrophoresis approaches we have reviewed are very likely to employ dynamic interactions of this type. For such a case, Ogston says, "It raises problems of explanation greater than any yet encountered."

On the basis of the limited experimental and analytical data, it is judged that the most critical step in the selected approach, separation, suffers "gaps" in knowledge in the following areas:

- o Effects of voltage gradient on enzyme mobility
- o Relationship of mobility to resolution
- o Local heating rates in gels
- o Convection rates in enzyme bands in gels
- o Effects of electrophoresis path length on resolution of isoenzymes
- o Relative effectiveness of large-, small-pore gel electrophoresis, and isoelectric-focussing.
- o Effects on total process, of variations in buffers, gel types, running time, voltage gradient, etc.

III.5.1.2 Other Elements in the Selected Approach

III.5.1.2.1 Sample Pre-Separation Storage

The biological materials are easily destroyed by heat, growth of micro-organisms and proteolytic enzymes present in impurities. It is expected that one of several existing techniques will suffice. It remains only to determine which will be most compatible with the sample to be separated in space operations.

III.5.1.2.2 Gel Stability During Launch

Gels must be capable of withstanding launch conditions. If they cannot, then the gels must be prepared in orbit. Preparation of gels, while a relatively simple operation on earth, could be a complex, time-consuming effort in orbit. No data exists at this time as to gel tolerance to launch "G's" and vibration.

III.5.1.2.3 Storage of Isoenzyme Bands in Gels After the Separation Operation

It must be determined whether gels, after running, can be stored for further work on earth. The elution of isoenzymes from gels is a comparatively straightforward process on earth. That element of the process, however, requires preservation of the pure bands of isoenzymes until return to earth. Effects of long (up to 2 weeks) storage on band broadening under handling loads is not known.

III.5.1.2.4 Equipment

Process equipment must be designed to withstand launch conditions, and be operable in zero "G". The applicability of ground-based apparatus to utilization in the space environment, while greatly desirable from the economic viewpoint, is, as yet, an unknown factor which must eventually be resolved.

III.5.1.2.5 Other Materials

Samples to be separated, buffers, preservatives, etc. must also be selected for their ability to withstand launch conditions, and be operable in orbit.

III.5.1.2.6 Total Process System

Assuming an experimental apparatus can be worked out, the usual problems of scale-up and design limitations must be addressed.

In summary, available information is insufficient to determine with certainty, whether large pore gel electrophoresis or isoelectric-focussing will yield the best product, and what are the best gels, buffers, path lengths, and temperature which must be selected for the products to be separated. There is, at present, little data on how these parameters vary with respect to optimum resolution, optimum sample stability, maximum loading, and ease of recovery of purified products. It has not been determined how long a gel containing isoenzyme bands, after running, can be stored without loss or resolution or denaturation of products, or whether gels can survive launch conditions. New equipment may have to be designed which can resist launch conditions; be useful for in-orbit separation via small pore and/or large pore electrophoresis and/or isoelectric-focussing; have closed buffer chambers and other special adaptations for using in zero gravity. It must also be determined whether a cooling system will be required.

III.5.2 EXPERIMENT REQUIREMENTS FOR VERIFICATION OF SELECTED APPROACH FOR SEPARATION OF ISOENZYMES

The identified knowledge "gaps" lead to the conclusion that certain experiments and calculations are required in order to demonstrate the feasibility of the selected approach; particularly that zero gravity conditions improve resolution of very similar biological macromolecules sufficiently to separate isoenzymes (and possibly other types of microheterogeneous molecules such as histones, immunoglobulins, and polynucleotides), many sets of which are not separable with present technology.

Specifically, the efforts required earliest are: Theoretical calculations of magnitude of convection and diffusion in various gels, and experimental verification of these calculations by measurements of band broadening vs. temperature

micro- and macrogradients in different gel types. Also we should know whether very long path lengths for increased separation of bands is effective under zero gravity - low convection conditions.

Another factor is the relationship of mobility to voltage gradient at very low voltage gradients, to determine if (1) molecules are better resolved at low voltage, (2) if molecules are spared from denaturation under low voltage conditions, and (3) if denaturation is less with large pore versus small pore gels. These early efforts do not require special equipment or zero gravity conditions for acquisition of these vital data.

A detailed breakdown has been prepared of the requirements for each experiment or test felt necessary to development of the final version of the approach to separation of isoenzymes. These requirements are summarized below.

The list of experiments falls into five groups shown in Figure III-31. The experiments in each group are related both in rationale and in timing. The group I experiments are designed to estimate the advantages of zero gravity separations. The factors studied are applicable to electrophoresis or isoelectric-focussing in general and are important for all separations, in contrast to the group II experiments. In group II, the selection of isoenzyme mixtures which will yield useful products is followed by identification of which general method (large or small pore gel electrophoresis or isoelectric-focussing) will give the best separation for each isoenzyme mixture, and which of the variables (buffers, gels, temperatures) will be best for <u>each</u> isoenzyme mixture. It should be emphasized that the factors in group II are optimized for each particular mixture to be separated as opposed to the factors discussed in group I, which apply to any separation. The group III experiments are designed to test the effects of

GROUP I.	PROCESS PHENOMENOLOGY
A	Relationship of Enzyme Mobilities to Voltage Gradient, at Low Voltage Gradients
B	Effects of Convective Disturbance on Resolution in Gel Electrophoresis
C	Use of Long Path Length to Improve Separation of Isoenzymes
D	Relation of Gel Length to Resolution in Isoelectric-Focussing
GROUP II.	SEPARATION EVALUATION
A	Best Ground Method of Separation of Selected Systems
B	Demonstration of Capability to Perform Preparative Scale Separations
GROUP III.	STUDY OF ENVIRONMENTAL FACTORS ON MATERIALS AND EQUIPMENT
A	Environmental Tests on Standard Equipment
B	Environmental Tests on Gels
C	Storage of Samples Without Denaturation
D	Storage of Post-Separation Products
GROUP IV.	DESIGN DATA AND VERIFICATION TESTING OF SPECIAL EQUIPMENT FOR ZERO GRAVITY
A	Separator Unit
B	Freezer-Cooling System Unit
C	Electrical Unit
GROUP V.	PROTOTYPE/PROOF TESTS
A	Short Term
B	Long Term
C	Prototype/Proof

Figure III-31. List of Verification Experiments or Tests for Separation of Isoenzymes

environmental factors, primarily in launch, but also in orbit and reentry, upon standard, commercial electrophoresis equipment, upon preformed gels, and upon biologicals. We would like to determine which materials, in their commonly available condition, will survive this testing, and which items will have to be modified for flight. Also, if modification is necessary, how to make these changes. Group IV investigates the flight worthiness of equipment specially designed and built for launch and orbital environments, while group V tests the effects of the space environment on the total separation system designed for automation and integration into the vehicle.

Specific requirements of experiments in these groups are summarized in Figures III-32 through III-47.

III.5.3 SUMMARY OF KNOWLEDGE GAPS, EXPERIMENTS AND TEST REQUIREMENTS OF SEPARATION OF ISOENZYMES

The previous discussion has dealt with the individual areas of unknowns in the selected approach, and subsequently briefly summarized the definition of each presently anticipated experiment and test to solve those unknowns.

Figure III-48 attempts to relate these unknowns, their required experiments and tests, and the requirements of those experiments and tests, all in one tabulation.

SEPARATION OF ISOENZYMES

PURPOSE:

TO EXAMINE THE MOBILITIES OF VARIOUS ENZYMES UNDER $\bar{V}$; TO DETERMINE IF THERE IS A MINIMUM V REQUIRED FOR MOBILITY, OR IF NEW SEPARATIONS ARE NOTED AT LOW V.

CONDITIONS:

STANDARD GROUND LABORATORY ENVIRONMENT AND CONDITIONS. VOLTAGE GRADIENTS ($<$ 10V/CM) VARIED FOR EACH RUN.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

A CLOSELY SPACED SET OF ISOENZYMES IS REQUIRED. TYPICAL SETS OF ISOENZYMES ARE (1) ESTERASES (READILY STAINED WITH INDOXYL ACETATE, P-NETROPHENYL ACETATE, COVER A LARGE RANGE OF MOBILITIES); (2) ALKALINE PHOSPHATASES (CLOSELY SPACED, INTERMEDIATE MOBILITY, EASILY STAINED WITH BROMOINDOXYL. N-ACETYL HEXOSAMINE).

EQUIPMENT, APPARATUS REQUIRED:

STANDARD ANALYTICAL ELECTROPHORETIC SEPARATOR, ELECTRONICS, AND COOLING DEVICES. A TYPICAL STANDARD COMMERCIAL SEPARATOR IS THE HOEFFER ELECTROPHORESIS UNIT #DE102. PHOTOGRAPHIC EQUIPMENT, MEASUREMENT EQUIPMENT, STAINING APPARATUS.

PROCEDURE OF TEST: (SEE FIGURE III-33)

THE SEPARATOR IS SET UP AS USUAL AND SAMPLES ARE APPLIED TO THE GEL TUBES (UP TO 12 DIFFERENT SAMPLES CAN BE RUN SIMULTANEOUSLY). THE ELECTRONICS WILL BE ADJUSTED FOR EACH RUN TO PROVIDE A PRE-SELECTED LOW V, AND THE STANDARD BUFFERS AND COOLING SYSTEMS ARE USED. A MUCH LONGER TIME THAN NORMAL WILL BE NECESSARY AT LOW $\bar{V}$, AND THE SEPARATION MAY RUN AS LONG AS 10 TO 15 HOURS. EACH EXPERIMENT OF 12 SAMPLES, THEREFORE, WILL REQUIRE ABOUT ONE DAY'S WORK, WITH THE SEPARATION STEP PROCEDING OVERNIGHT. THE ANALYSIS OF THE SEPARATIONS IN THE GELS IS PERFORMED BY USING THE WELL KNOWN STAINS AND ENZYME SUBSTRATES TO MARK THE INDIVIDUAL BANDS. STAINING AND PRESERVING THE GELS TAKES ONLY A FEW MINUTES OF OPERATOR ATTENTION, ALTHOUGH THE PROCESS MAY REQUIRE 1 TO 2 DAYS. THE GELS WILL BE PRESERVED IN 8% ACETIC ACID.

Figure III-32. Summary Definition of Requirements for Verification Experiments or Tests, IA - Relationship of Enzyme Mobilities to Voltage Gradient, at Low Voltage Gradients

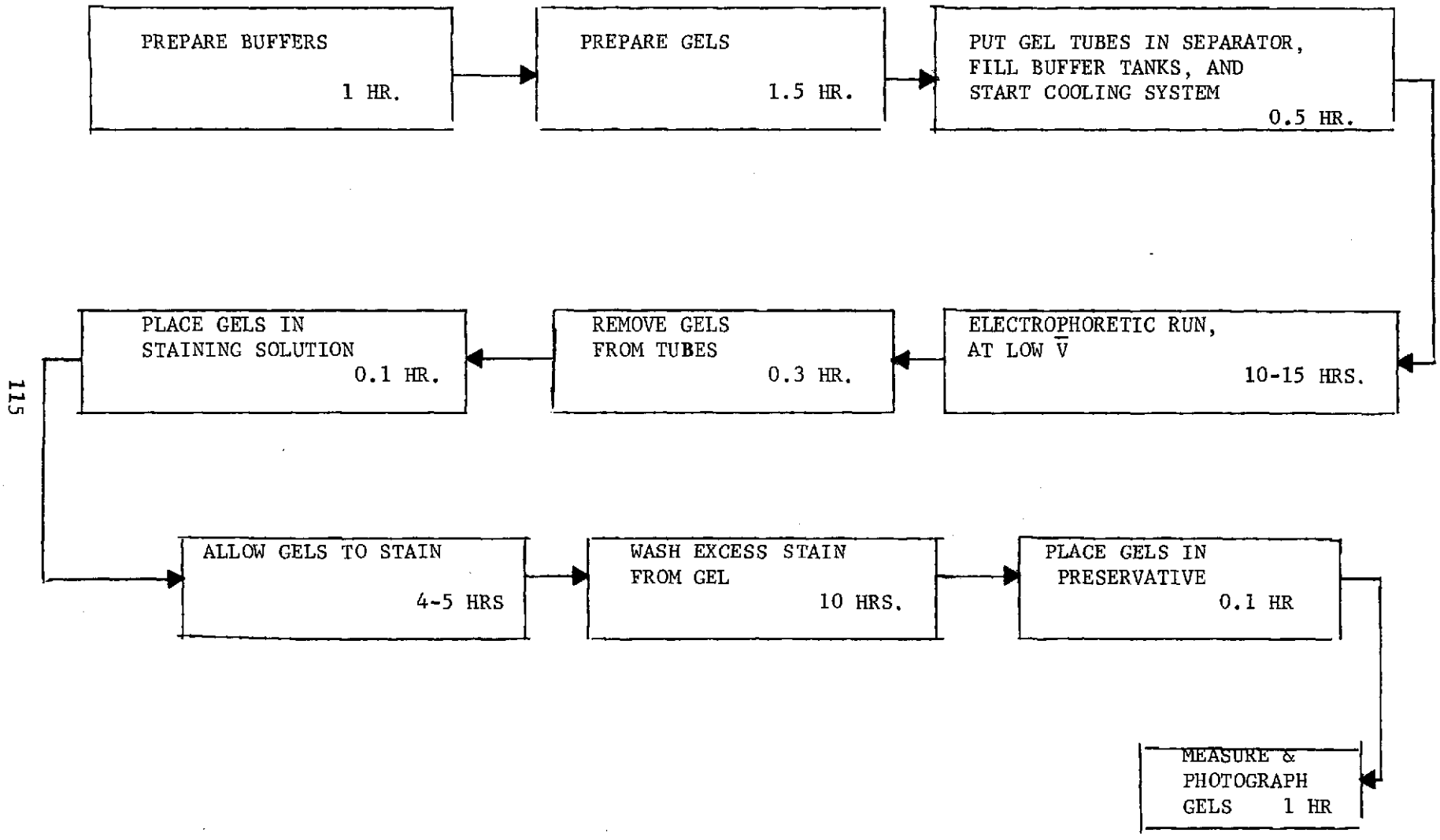

Figure III-33. Steps for a Typical Electrophoresis Run

SEPARATION OF ISOENZYMES

PURPOSE:

TO QUANTIFY CONVECTION CURRENT EFFECTS ON RESOLUTION OF ISOENZYME BANDS.

CONDITIONS:

STANDARD GROUND ENVIRONMENT AND CONDITIONS. TEMPERATURE GRADIENTS VARIED FOR EACH RUN.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

AS IN IA.

EQUIPMENT, APPARATUS REQUIRED:

AS IN IA, PLUS HEATING ELEMENTS TAILORED TO THE SEPARATOR TUBES, CONTROLS, FOR 10° - 30°C INCREASES IN GEL TEMPERATURE. COOLING ELEMENTS TAILORED TO THE SEPARATOR TUBES, HIGH ACCURACY THERMOCOUPLES WITH CONTROLS TO MAINTAIN MINIMUM TEMPERATURE GRADIENTS.

PROCEDURE OF TEST:

ISOENZYMES WILL BE SEPARATED IN STANDARD SEPARATOR. UNSTAINED BANDS WILL BE SUBJECTED TO CONVECTIVE FORCES BY CONTROLLED HEATING AND COOLING OF THE GELS IN LOCALIZED AREAS TO CREATE AND MINIMIZE CONVECTION MIXING. THE GELS WILL THEN BE STAINED IN THE USUAL MANNER, AND COMPARED TO CONTROL GROUPS, FOR THE AMOUNT OF BAND DISTORTION BROADENING.

VARIOUS GEL TYPES, SUCH AS LARGE PORE AND SMALL PORE WILL BE TESTED TO ASSESS THEIR EFFECTS ON CONVECTIVE MIXING.

EACH RUN OF 12 TUBES WILL REQUIRE 2 DAYS; 10 RUNS WILL BE SUFFICIENT.

Figure III-34. Summary Definition of Requirements for Verification Experiments or Tests, IB - Effects of Convective Disturbance on Resolution in Gel Electrophoresis

SEPARATION OF ISOENZYMES

PURPOSE:

TO DETERMINE WHETHER LONG PATH LENGTH WILL IMPROVE RESOLUTION OF ISOENZYMES WITH VERY SIMILAR MOBILITIES.

CONDITIONS:

AS IN IA.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

AS IN IA.

EQUIPMENT, APPARATUS REQUIRED:

AS IN IA, WITH SPECIAL LONG PATH GEL TUBES, WHICH WILL FIT INTO STANDARD SEPARATOR. COILED TUBE SHOULD BE SATISFACTORY.

PROCEDURE OF TEST:

STANDARD ELECTROPHORESIS PROCEDURE USING ISOENZYME SETS AS IN IA. RUNS WILL TAKE SUBSTANTIALLY LONGER THAN NORMAL, ESTIMATE ABOUT 6 HOURS. COILED TUBES TREATED WITH A WETTING AGENT (KODAK PHOTOFLOW) OR SILICONIZED TO GREATLY REDUCE ADHESION OF THE GEL TO THE GLASS, MAKING IT EASY TO FORCE THE GEL OUT OF THE TUBE. AFTER GEL IS REMOVED, IT IS STAINED IN THE USUAL MANNER TO VISUALIZE THE BANDS. EACH TOTAL EXPERIMENT WILL REQUIRE 1 TO 2 DAYS, BUT ONLY 1 OR 2 SPECIAL LONG PATH TUBES CAN BE USED PER SEPARATION RUN; WE REQUIRE 10 TO 15 RUNS.

Figure III-35. Summary Definition of Requirements for Verification Experiments or Tests, IC - Use of Long Path Length to Improve Separation of Isoenzymes

SEPARATION OF ISOENZYMES

PURPOSE:

TO DETERMINE WHETHER A LONG GEL LENGTH WILL RESULT IN BETTER RESOLUTION IN GEL ISOELECTRIC FOCUSSING.

CONDITIONS:

AS IN IA.

SAMPLES, SPECIMENS, ITEMS TO BE TESTED:

AS IN IA.

EQUIPMENT, APPARATUS REQUIRED:

AS IN IA, WITH SPECIAL LONG PATH TUBES AS IN IC.

PROCEDURE:

SAME AS IC, WITH ADDITION OF AN AMPHOLYTE MIXTURE TO THE MONOMER MIXTURE BEFORE POLYMERIZATION. SAMPLE ADDED TO THE TOP OF THE GEL COLUMN OR MIXED WITH THE MONOMER SOLUTION BEFORE POLYMERIZATION. RUNNING TIME WILL BE QUITE LONG - 10 TO 12 HOURS, BUT OTHER STEPS WILL TAKE THE SAME TIME AS IN IA. 10 TO 15 RUNS WILL BE REQUIRED.

Figure III-36. Summary Definition of Requirements for Verification Experiments or Tests,ID - Relation of Gel Length to Resolution in Isoelectric Focussing

SEPARATION OF ISOENZYMES

PURPOSE:

TO FIND THE OPTIMUM METHOD OF RESOLVING THE SPECIFIC ISOENZYME OF INTEREST.

CONDITIONS:

AS IN IA. STANDARD METHODS INITIALLY, MODIFICATIONS LATER. FOR EACH ISOENZYME SYSTEM, THE VARIABLES WILL BE ALTERED, ONE AT A TIME, AS SHOWN IN FIGURE III-38.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

LARGE, SMALL PORE GELS, ISOELECTRIC FOCUSSING EQUIPMENT, VARIOUS BUFFERS. SAMPLES OF SERUM, TISSUE, ETC. FROM PATIENTS. FOR THIS ANALYTICAL WORK, SEVERAL MILLILITERS OF BLOOD, SEVERAL GRAMS OF TISSUE WILL BE SUFFICIENT.

EQUIPMENT, APPARATUS REQUIRED:

AS IN IA, WITH LATER POSSIBILITY OF MODIFICATIONS.

PROCEDURE:

FIRST PRELIMINARY PURIFICATION. SUCH PURIFICATION MUST BE FOLLOWED AT EACH STEP BY ASSAYS OF BIOLOGICAL ACTIVITY, AND BY ELECTROPHORESIS TO ESTIMATE THE DEGREE OF PURIFICATION ACHIEVED, OR TO DISCOVER IF THE WANTED MATERIAL WAS DENATURED OR LOST. THE PARTIALLY PURIFIED MATERIAL MUST BE CAPABLE OF STORAGE. THE ESTIMATED TIME FOR PRELIMINARY PURIFICATION FOR EACH ISOENZYME SYSTEM IS TWO MONTHS. THE ESTIMATED TEST TIME FOR DETERMINATION OF BEST SEPARATION METHOD WILL BE ONE MONTH FOR EACH SYSTEM, AT 1 TO 2 DAYS PER RUN.

Figure III-37. Summary Definition of Requirements for Verification Experiments or Tests, IIA - Best Ground Method of Separation of the Selected Separation Systems

GENERAL METHOD	$\bar{V}$	PATH LENGTH	BUFFER pH	TEMP OF GEL
LARGE PORE, G.E. (STANDARD)	10 V/CM	10 CM	9.0	5°C
MODIFIED	VARIABLE	10 CM	9.0	5°C
MODIFIED	10 V/CM	VARIABLE	9.0	5°C
MODIFIED	10 V/CM	10 CM	VARIABLE	5°C
SMALL PORE G.E. (STANDARD)	10 V/CM	10 CM	9.0	5°C
MODIFIED	VARIABLE	10 CM	9.0	5°C
MODIFIED	10 V/CM	VARIABLE	9.0	5°C
ISOEL. FOCUS (STANDARD)	10 V/CM	10 CM	AMPHOLYTES pH 2-8	5°C

Figure III-38. Test Variables, Isoenzyme Separation Test, IIA

SEPARATION OF ISOENZYMES

PURPOSE:

TO DETERMINE BEST SCALE UP METHOD FOR EACH ISOENZYME SYSTEM.

CONDITIONS:

AS IN IA.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

PREPARATIVE-SCALE SEPARATOR, PERIPHERAL EQUIPMENT, SELECTED BUFFERS, VOLTAGE GRADIENT. SAMPLES DETERMINED BY IIA FROM HOSPITALS, CLINICS, AND PHYSICIANS. ~1 LITER OF BLOOD OR SERUM, OR WHOLE ORGANS.

EQUIPMENT, APPARATUS REQUIRED:

AS IN IA. COMMERCIAL PREPARATIVE SCALE SEPARATORS FOR EARLY SCALE UP. LATER, MODIFIED SEPARATORS, INCLUDING SPECIAL LONG PATH LENGTH SEPARATORS.

PROCEDURE:

USE PREPARATIVE SCALE SEPARATORS AND THE BEST METHODOLOGY FOR EACH SYSTEM AS DETERMINED IN IIB. ONE SAMPLE PER RUN, THE RUNS TAKING ABOUT 1 DAY EACH. METHODS OF REMOVING THE SAMPLES FROM THE GEL WILL BE STUDIED; REMOVE THE GEL FROM ITS HOLDER, SLICE AND ELUTE BANDS, USE THE CONTINUOUS ELUTION METHOD. THE RECOVERED SAMPLES MUST BE EXAMINED FOR HOMOGENIETY BY (1) BIOLOGICAL ASSAYS; (2) RUNNING ON ANALYTICAL SCALE ELECTROPHORESIS. EACH SAMPLE WILL REQUIRE ONE DAY FOR SEPARATION, ONE DAY FOR ELUTION AND CONCENTRATION, AND ONE TO TWO DAYS FOR ANALYSIS.

Figure III-39. Summary Definition of Requirements for Verification Experiments or Tests, IIB - Demonstration of Capability to Perform Preparative Scale Separations

SEPARATION OF ISOENZYMES

PURPOSE:

TO DETERMINE THE STRUCTURALLY WEAK POINTS OF STANDARD EQUIPMENT USED IN ELECTROPHORESIS, ESPECIALLY THE SEPARATOR UNIT.

CONDITIONS:

TYPES AND MAGNITUDES OF LOADINGS EXISTING DURING LAUNCH; SIMULATED SHOCK AND VIBRATION TESTS ON CENTRIFUGE.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

SEVERAL MODELS OF PREPARATIVE SCALE SEPARATORS, POWER SUPPLIES AND FREEZER/COOLER UNITS WILL BE TESTED, SPECIFIC GEL AND ISOENZYME SET.

EQUIPMENT, APPARATUS REQUIRED:

SHOCK, VIBRATION TESTERS, PROGRAMMABLE CENTRIFUGE, HEATERS.

SPECIAL SUPPORT REQUIRED:

INSTRUMENTATION, DATA HANDLING EQUIPMENT FOR ABOVE.

PROCEDURE:

MOUNT SEPARATORS IN TEST EQUIPMENT. SUBJECT TO LOADINGS FOR LAUNCH DURATION. VARY EQUIPMENT ORIENTATION; VARY SHOCK, VIBRATION ISOLATION. ESTIMATE 15 TO 20 RUNS, ~10 MINUTES PER RUN.

Figure III-40. Summary Definition of Requirements for Verification Experiments or Tests, IIIA - Environmental Tests and Standard Equipment

SEPARATION OF ISOENZYMES

PURPOSE:

TO EXAMINE THE RESISTANCE OF GELS TO LAUNCH CONDITIONS.

CONDITIONS:

AS IN IIIA.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

SEVERAL GEL TYPES INCLUDING LARGE AND SMALL PROEGELS, WITH VARIOUS CROSSLINKERS, METHYLENE BISACRYLAMIDE, HEXAMETHYLENE BISACRYLAMIDE, AND VARYING SOLIDS CONCENTRATION IN TUBES OF SPECIAL DESIGN. ALTERNATIVES; (1) ENDS OF THE TUBE CONSTRICTED; (2) PLUGS INSERTED IN THE ENDS OF THE TUBE; (3) GEL FROZEN.

EQUIPMENT, APPARATUS REQUIRED:

AS IN IIIA.

PROCEDURE:

TUBES WITH GEL SPECIMENS TO BE MOUNTED IN CENTRIFUGE AND VIBRATED WHILE SUBJECTED TO LAUNCH TRAJECTORY LOADINGS. THE GELS SUBSEQUENTLY TESTED FOR LENGTH OF TIME THEY REMAIN IN GOOD CONDITION.

ABOUT ONE WEEK TO PREPARE SPECIAL TUBES, PLUGS, ETC. AND TO PREPARE VARIOUS BUFFERS AND SOLUTIONS. EACH GEL TYPE TO BE USED WITH THE THREE DESCRIBED METHODS. TESTING OF SEPARATION EFFICACY BY RUNNING STANDARD SAMPLES, AS IN IA, OF ISOENZYMES AFTER VARYING STORAGE PERIODS. GELS WILL BE TESTED AS IN IIIA. ESTIMATE 15 TO 20 RUNS, 30 MINUTES PER RUN.

Figure III-41. Summary Definition of Requirements for Verification Experiments or Tests, IIIB - Environmental Tests on Gels

SEPARATION OF ISOENZYMES

PURPOSE:

TO DETERMINE INITIAL SAMPLE STORAGE METHOD FOR AT LEAST A SEVERAL WEEK PERIOD AND TO RECONSTITUTE WITHOUT DEGRADATION.

CONDITIONS:

STANDARD GROUND CONDITIONS.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

FROM IIA AND IIB, LYOPHILIZED, BUFFERED, REFRIGERATED.

EQUIPMENT, APPARATUS REQUIRED:

AS IN IA PLUS ENVIRONMENTAL CHAMBER TO SIMULATE ALL PREFLIGHT, FLIGHT CONDITIONS, RECONSTITUTION EQUIPMENT.

SPECIAL SUPPORT REQUIRED FOR TEST:

BIOLOGICAL ASSAY OF SPECIMENS.

PROCEDURE:

AFTER PRELIMINARY PURIFICATION (IIB), THE SAMPLES WILL BE STORED BY THE THREE TYPICAL METHODS AND WILL BE SAMPLED ONCE A WEEK FOR UP TO 8 WEEKS. SAMPLING WILL CONSIST OF BIOLOGICAL ASSAYS AND ELECTROPHORESIS TO DETERMINE THE AMOUNT OF DENATURATION. EACH ASSAY WILL TAKE ONE DAYS WORK, AND WILL BE PERFORMED FOR THE SEVERAL ENZYME SYSTEMS CHOSEN IN IIA AND IIB.

Figure III-42. Summary Definition of Requirements for Verification Experiments or Tests, IIIC - Storage and Reconstitution of Samples Without Denaturation

SEPARATION OF ISOENZYMES

PURPOSE:

TO DEVELOP STORAGE AND HANDLING METHOD FOR THE SEPARATED PRODUCTS WITH STABILITY FOR AT LEAST FOUR WEEKS.

CONDITIONS:

STANDARD GROUND CONDITIONS.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

AS IN IIIC, AFTER ELECTROPHORETIC SEPARATION.

EQUIPMENT, APPARATUS REQUIRED:

AS IN IA, IIIC, POST FLIGHT SIMULATION.

SPECIAL SUPPORT REQUIRED FOR TEST:

CENTRIFUGING AND VIBRATION OF PRODUCTS PRIOR TO TEST TO SIMULATE RECOVERY LOADINGS. BIOLOGICAL ASSAY OF PRODUCTS.

PROCEDURE:

THE ENZYME SYSTEMS WILL BE SEPARATED ON AN ANALYTICAL SCALE APPARATUS, STORED BY THE METHODS MENTIONED IN IIIC AND TESTED ONCE A WEEK AS DESCRIBED IN IIIC. THE MATERIAL WILL ALSO BE TESTED AFTER STORAGE IN FROZEN GEL. ONE HOUR LOAD SIMULATION PER RUN, 8 WEEKS PER ASSAY.

Figure III-43. Summary Definition of Requirements for Verification Experiments or Tests, IIID - Post-Separation Storage and Handling of Products

SEPARATION OF ISOENZYMES

PURPOSE:

DESIGN TESTING OF SEPARATOR UNIT, BUFFER STORAGE AND TRANSFER, SPECIMEN LOADING, COOLING SYSTEM, BUBBLE CONTROL.

CONDITIONS:

AS IN IIIA, PLUS ZERO "G" TESTING BY PARABOLIC AIRPLANE FLIGHTS, SOUNDING ROCKETS, ORBITING SPACECRAFT, SHUTTLE.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

SPECIALLY DESIGNED SEPARATOR UNITS. ESTIMATED WEIGHT = 500-1000g AND ESTIMATED VOLUME = 20,000 CM^3.

EQUIPMENT, APPARATUS REQUIRED:

AS IN IIIA, KC-135, SOUNDING ROCKETS, SPACECRAFT, SHUTTLE. PHOTOGRAPHIC AND ELECTRONIC DATA ACQUISITION, COMMUNICATIONS. PROCESS INSTRUMENTATION.

SPECIAL SUPPORT REQUIRED FOR TEST:

PREFLIGHT HANDLING AND CHECKOUT. ON-ORBIT AUTOMATED AND MANUAL OPERATIONS. POST-FLIGHT HANDLING, STORAGE. ENVIRONMENT INSTRUMENTATION. TECHNICIAN MONITORING, MAINTAINING SHUTTLE TESTS.

PROCEDURE:

IN ADDITION TO TESTING AS IN IIIA, THE OPERATIONS OF FILLING AND EMPTYING BUFFER TANKS, REMOVING BUBBLES FROM BUFFERS, CHANGING GEL TUBES WILL BE STUDIED IN A ZERO "G" SITUATION, BOTH IN AUTOMATED AND MANUAL MODES FOR GROUND TESTING, 20 MINUTES PER RUN, 15 TO 20 RUNS.

FOR KC-135, 30 SECONDS PER RUN, 40 TO 100 RUNS. FOR SOUNDING ROCKETS, 1 TO 10 MINUTES PER RUN, 1 TO 2 RUNS. FOR SPACECRAFT, 1 DAY PER RUN, 1 OR 2 RUNS. FOR SHUTTLE, 1 DAY PER RUN, 1 OR 2 RUNS.

Figure III-44. Summary Definition of Requirements for Developments Tests, IVA - Design Testing of Separator Unit

SEPARATION OF ISOENZYMES

PURPOSE:

TO DESIGN TEST A FREEZER UNIT FOR SUPPORT OF SEPARATION OF ISOENZYMES IN SPACE.

CONDITIONS:

AS IN IVA, NO SOUNDING ROCKET.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

FREEZER UNITS. ESTIMATED WEIGHT = 10 KG. ESTIMATED VOLUME = 0.1 TO 0.5 M^3.

EQUIPMENT, APPARATUS REQUIRED:

AS IN IVA, NO SOUNDING ROCKET.

SPECIAL SUPPORT REQUIRED:

AS IN IVA.

PROCEDURE:

LOAD SAMPLES, FINISHED GELS, AND SEPARATOR UNITS; SUCH THAT CONTENTS ARE HELD FIRMLY IN PLACE; EASILY REMOVED; PROTECTED FROM SHOCK, VIBRATION, "G" LOADS; VARIOUS ORIENTATION. OPERATE. RECOVER. CHECK FOR DAMAGE, CHANGES, ETC. TIMING AS IN IVA, NO SOUNDING ROCKET.

Figure III-45. Summary Definition of Requirements for Development Tests, IVB - Design Testing of Freezer-Cooling System Unit

SEPARATION OF ISOENZYMES

PURPOSE:

TO DESIGN TEST A SUITABLE ELECTRICAL POWER UNIT.

CONDITIONS:

AS IN IVA, NO SOUNDING ROCKET.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

ELECTRICAL POWER UNIT FOR SPACE SEPARATION OF ISOENZYMES.
ESTIMATED WEIGHT = 5 KG. ESTIMATED VOLUME = 0.1 M^3.

EQUIPMENT, APPARATUS REQUIRED:

AS IN IVA, NO SOUNDING ROCKET, NO PHOTOGRAPHIC EQUIPMENT.

SPECIAL SUPPORT REQUIRED:

AS IN IVA.

PROCEDURE:

OPERATE FROM ON-BOARD POWER SUPPLY AT LEVELS REQUIRED FOR SEPARATION, FOR MAXIMUM DURATION, OR FLIGHT DURATIONS OF IVA. RECORD PERFORMANCE. RECOVER, IF POSSIBLE, FOR PHYSICAL EXAMINATION.

Figure III-46. Summary Definition of Requirements for Development Tests, IVC - Design Testing of the Electrical Unit

SEPARATION OF ISOENZYMES

PURPOSE:

TO PROOF TEST THE COMPLETE ISOENZYME SEPARATION SYSTEM, INCLUDING THE AUTOMATION EQUIPMENT. TO FINALIZE THE INTEGRATION OF ALL UNITS OF THE SEPARATION SYSTEM, THE INTEGRATION OF THE SEPARATION SYSTEM WITH THE SUPPORT EQUIPMENT, AND WITH THE SHUTTLE.

CONDITIONS:

SPACE SHUTTLE LABORATORY MODULE, ORBITAL ENVIRONMENT.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

COMPLETE PREPARATIVE-SCALE ISOENZYME SEPARATION SYSTEM, SAMPLES AS IN IIB. ESTIMATED WEIGHT $\backsim$20 KG. ESTIMATED VOLUME $\backsim$.25 TO .27 M^3.

EQUIPMENT, APPARATUS REQUIRED:

SPACE SHUTTLE LABORATORY, ENVIRONMENTAL AND PROCESS INSTRUMENTATION, ELECTRIC POWER AND OTHER UTILITIES SERVICES.

SPECIAL SUPPORT REQUIRED:

ON-BOARD TEST OPERATOR, TWO WAY COMMUNICATIONS WITH TEST CONTROLLER ON GROUND, PHOTOGRAPHY.

PROCEDURE:

LAUNCH IN MODE ESTABLISHED FROM EARLIER TESTS, (FORMED GEL, PRE-LOADED SPECIMENS OR FORM GEL AND LOAD IN ORBIT). OPERATOR CHECK-OUT, WARM UP, SYSTEM, AND INITIATE AUTOMATED PROCESS, OBSERVE PROCESS STEPS, CARRY OUT NECESSARY FINE TUNING AND MANUAL FUNCTIONS. MAINTAIN COMMUNICATIONS WITH GROUND INVESTIGATOR, PHOTOGRAPH ANOMALIES AND FINAL RESULTS. ESTIMATE ONE DAY PER RUN, 5 TO 10 RUNS REQUIRED.

Figure III-47. Summary Definition of Requirements for Development Tests, V - Separation System Prototype/Proof and Integration Tests

KNOWLEDGE GAPS	EXPERIMENTS AND VERIFICATION TESTS	EXPERIMENT AND TEST REQUIREMENTS (SUMMARY)
RELATIONSHIP OF ENZYME MOBILITY IN GELS TO VOLTAGE GRADIENT (GRADIENTS < 10 V CM) AND RELATIONSHIP OF MOBILITY TO ISOENZYME RESOLUTION	TESTS OF SPECIFIC ENZYMES IN VARIOUS GELS AT SPECIFIC VOLTAGE GRADIENTS, AND ON A CLOSELY SPACED SET OF ISOENZYMES AT ESTABLISHED MOBILITIES	STANDARD GROUND LAB, STANDARD ELECTROPHORETIC SEPARATOR (TYPICALLY, HOEFFER DE(02) AND PERIPHERALS, CONTROLLED VOLTAGE GRADIENTS (< 10V CM), ISOENZYME SETS (ESTERASES, ALKALINE PHOSPHATASES, HEXOSAMINIDASES), PHOTOGRAPHIC AND MEASUREMENT EQUIPMENT, ANALYTICAL STAINING APPARATUS, 10–15 HOURS PER RUN, MANUAL LOADING, INITIATION, ADJUSTMENTS , TERMINATION, SAMPLE TRANSFER, AUTOMATED RUNNING. LATER SPACECRAFT EXPERIMENTS, NEW SEPARATOR SYSTEM, SIMILAR EQUIPMENT, 1 DAY PER RUN, AUTOMATED. PROCESS AND ENVIRONMENT INSTRUMENTATION, PHOTOGRAPHY. TELEMETRY OF DATA. RECOVERY OF SPECIMENS, PHOTOGRAPHS DESIRED
MEASUREMENT OF LOCAL HEATING WITHIN A GEL DURING ELECTROPHORESIS AND RATE OF CONVECTIVE DISTURBANCE OF ENZYME BANDS IN GELS	TESTS USING SMALL TEMPERATURE SENSORS IN GEL DURING ELECTROPHORESIS AT VARIOUS VOLTAGE GRADIENTS TO DETERMINE HEATING AND CONVECTIVE EFFECTS ON SEPARATED BANDS	STANDARD GROUND LAB, EQUIPMENT AS ABOVE PLUS CONTROLLABLE (10–30°C) HEATERS TAILORED TO ELECTROPHORESIS TUBES, THERMOCOUPLES IN GEL. 2 DAYS PER RUN. LATER SPACECRAFT EXPERIMENTS WITH NEW SEPARATOR SYSTEM, SIMILIAR EQUIPMENTS, 1 DAY PER RUN. PROCESS AND ENVIRONMENT INSTRUMENTATION, PHOTOGRAPHY. TELEMETRY OF DATA. RECOVERY OF SPECIMENS, PHOTOS DESIRED.
RELATIONSHIP OF PATH LENGTH TO RESOLUTION OF ISOENZYMES	TESTS ON A CLOSELY SPACED SET OF ISOENZYME S AT VARIOUS PATH LENGTHS	STANDARD LAB, MODIFIED STANDARD ELECTROPHORETIC SEPARATOR TO ACCOMMODATE LONGER (PROBABLY HELICAL) TUBES, SELECTED VOLTAGE GRADIENTS, SELECTED ISOENZYME SYSTEM, ANALYTICAL STAINING APR, PHOTOGRAPHIC AND MEASUREMENT EQUIPMENT, 1 DAY PER RUN. MANUAL AND AUTOMATED TASKS AS ABOVE. LATER, SPACECRAFT TESTS OF NEW SEPARATOR SYSTEM WITH SELECTED TUBE LENGTH, SELECTED VOLTAGE GRADIENT, SELECTED ISOENZYME SYSTEM, PHOTOGRAPHIC AND MEASUREMENT EQUIPMENT, OTHER EQUIPMENT AND INSTRUMENTATION FOR GROUND–BASED ANALYSIS, 1 DAY PER RUN. AUTOMATED. TELEMETRY OF INSTRUMENT DATA, RECOVERY OF EQUIPMENTS AND SPECIMENS DESIRED.
BEST SEPARATION METHOD	TESTS OF SEVERAL SPECIFIC ISOENZYME SYSTEMS TO COMPARE LARGE AND SMALL PORE GEL AND ISOELECTRIC FOCUSSING SYSTEMS, VARYING BUFFER, GEL TYPES, RUNNING TIME, VOLTAGE GRADIENT, ETC.	STANDARD GROUND LAB, STANDARD ELECTROPHORETIC SEPARATOR (INITIALLY, MODIFIED OR NEW LATER), EQUIPMENT TO CARRY OUT LARGE AND SMALL PORE ELECTROPHORESIS (SEVERAL GELS) ISOELECTRIC FOCUSSING, VARIOUS BUFFERS, VOLTAGE REGULATION, PHOTOGRAPHIC AND MEASUREMENT EQUIPMENT, ANALYTICAL STAINING APPARATUS, 1 DAY PER RUN. MANUAL AND AUTOMATED TASKS AS ABOVE.
SCALE–UP PARAMETERS AND DESIGN LIMITS	TESTS OF PREPARATIVE SCALE EQUIPMENT TO ASSESS SCALE–UP EFFECTS ON OHMIC HEATING, CONVECTION, RESOLUTION	STANDARD GROUND LAB, NEW LARGE SCALE SEPARATOR, SELECTED BUFFER, VOLTAGE GRADIENT, PERIPHERAL EQUIPMENT FOR SELECTED SEPARATION METHOD, TEST INSTRUMENTATION, ANALYTICAL STAINING APPARATUS, PHOTOGRAPHIC AND MEASUREMENT EQUIPMENT. 1 DAY PER RUN. MANUAL AND AUTOMATED TASKS AS ABOVE.
EFFECTS OF LAUNCH ENVIRONMENTS ON EQUIPMENT, SPECIMENS, GELS, BUFFERS, ETC.	ENVIRONMENTAL TESTS (E.G., SHOCK, TEMP., VIBRATION, ACCELERATION, ETC.)	PROGRAMMABLE CENTRIFUGE, GONDOLA–MOUNTED VIBRATION, SHOCK, HEATING EQUIPMENT, VARIOUS GELS, OTHER SEPARATION–RELATED COMPONENTS, SPECIMENS, TEMPERATURE, VIBRATION, LOAD–MEASURING INSTRUMENTATION, 30 MINUTES PER RUN, AUTOMATED.
BEST METHOD OF PRESERVATION OF SPECIMENS PRELAUNCH	TESTS OF PRESERVATIVE ADDITIVES, FORM OF PRESERVATION AND ENVIRONMENTS	ENVIRONMENTAL CHAMBER (TEMP, HUMIDITY PRESSURE) CONTROLLABLE FOR VARIOUS PRE–FLIGHT CONDITIONS. LYOPHILIZED, BUFFERED, REFRIGERATED SPECIMENS, BIOLOGICAL ASSAY EQUIPMENT, UP TO 8 WEEKS PER RUN. AUTOMATED, WITH MANUAL ASSAY.
ORBITAL PREPARATION FOR SEPARATION	TECHNIQUES TO STORE AND RECONSTITUTE FOR SEPARATION	STANDARD GROUND LAB, SAMPLES FROM LYOPHILIZED, BUFFERED, REFRIGERATED SPECIMENS, RECONSTITUTION EQUIPMENT (HYDRATION, HEATING), BIOLOGICAL ASSAY EQUIPMENT, UP TO 1 DAY PER TEST. MANUAL. LATER, SHUTTLE–BASED, TESTS; SPECIMENS OF SELECTED PRESERVATION METHODS, SELECTED RECONSTITUTION EQUIPMENT, 1 DAY PER TEST, RECOVERY OF EQUIPMENT, SPECIMENS, TELEMETRY OF DATA. BIO–ASSAY PERFORMED ON GROUND MANUAL.
BEST METHOD OF PRESERVATION OF SEPARATED ISOENZYMES TO MAINTAIN BIOLOGICAL LIFE AND PURITY, AND PHYSICAL SEPARATION AND FOR POST–FLIGHT BIOLOGICAL ADEQUACY	HANDLING TECHNIQUES, PRESERVATIVES, FORM OF PRESERVATION AND ENVIRONMENTS TO PRESERVE TEST PRESERVED PRODUCTS FOR RECOVERY ENVIRONS RECONSTITUTE PRESERVED MATERIALS AFTER EXPOSURE TO RECOVERY ENVIRONMENTS DEMONSTRATE BIOLOGICAL ADEQUACY OF ISOENZYMES	STANDARD GROUND LAB FOR LYOPHILIZATION, BUFFERING, REFRIGERATING OF SEPARATED ISOENZYMES, PROGRAMMABLE CENTRIFUGE WITH VIBRATOR TO SIMULATE RECOVERY LOADING. ~ 1 HOUR PER RUN. AUTOMATED BIO–ASSAY EQUIPMENT FOR DETERMINATION OF BIO–QUALITY. ~ 8 WEEKS PER RUN. MANUAL.
EFFECTS OF SPACE ENVIRONMENTS ON THE SELECTED SEPARATION PROCESS, EQUIPMENT DESIGN.	DETERMINE THE SPACE ENVIRONMENT PERFORMANCE OF SELECTED PROCESS EQUIPMENT DESIGNS. ZERO–G AIRCRAFT AND SOUNDING ROCKET TESTS OF PROCESS EQUIPMENT ORBITAL TEST OF PROCESS EQUIPMENT PROTOTYPE PRODUCTION OPERATIONS	SHORT TERM ZERO "G" TESTS OF BUFFER STORAGE AND TRANSFER, SPECIMEN INJECTION, COOLING SYSTEM, ETC. DESIGNS (MATERIALS BUBBLE CONTROL, ETC.) – ZERO "G" AIRCRAFT SOUNDING ROCKET. ENVIRONMENT INSTRUMENTATION, PHOTOGRAPHY. RECOVERY OF RECORDED DATA, EQUIPMENTS. ~ 1 TO 10 MINUTES PER RUN. AUTOMATED, LONG TERM ZERO "G" TESTS OF CONTROLS, GEL STABILIZATION, AUTOMATION EQUIPMENT, ETC. ~ 1 DAY PER RUN, AUTOMATED. ORBITAL DEMONSTRATION OF FULL–SCALE PROCESS EQUIPMENT. ~ 1 DAY PER RUN. AUTOMATED, WITH TECHNICIAN OBSERVING, ENVIRONMENTAL AND PROCESS INSTRUMENTATION. PHOTOGRAPHY. RECOVERY OF EQUIPMENT, SPECIMENS, TELEMETRY OF DATA.

Figure III-48. Definition of Requirements for Experiments to Verify Selected Approach for Separation of Isoenzymes

III.6 DEFINITION OF REQUIREMENTS FOR EXPERIMENTS TO VERIFY SELECTED APPROACH FOR PROCESSING TRANSPARENT OXIDES

III.6.1 CRITICAL ELEMENTS IN SELECTED APPROACH FOR PROCESSING OF TRANSPARENT OXIDES, AND RELATED KNOWLEDGE "GAPS"

Of the four products under study in Phase II, this area possesses the least available information for forecasting the success with which it may be accomplished. Ground laboratory experiments aimed at producing amorphous forms of the oxides considered herein have produced extremely small specimens with the desired properties. The technique utilized in those experiments, cooling of molten oxides in free fall, however, could not be scaled-up in ground-based facilities to achieve products of needed size, and other ground-based techniques have, so far, proved unsatisfactory.

The selected best approach defined in Section III.2.3 is expected to overcome the homogeneous and heterogeneous nucleation which occurs in present ground-based techniques of useful size. The fact that homogeneous nucleation has not been observed in silica glass leads to the expectation that it might also not occur in the proposed single oxide glasses; Alumina, Zirconia and Yttria. Heterogeneous nucleation is also expected to be avoided in the defined approach through the use of high purity initial materials and further purification steps in the defined process, thus eliminating contaminants as a source of nucleation. Most important, the proposed containerless melting and resolidification eliminates crucible walls as a nucleation surface.

Finally, even if nucleation cannot be completely avoided in the selected process, the better control and uniformity of cooling possible via containerless processing is expected to produce heretofore unobtainable crystalline structures in the materials under study, offering unique properties of potential value in technical applications.

The following elements of the defined best approach for processing Transparent Oxides are considered critical steps, and are subject to unknowns in the state-of-the-art, as discussed within the body of each paragraph.

III.6.1.1 Cooling

The cooling step in the selected process is the most critical. Assuming that all preceding steps culminate in desired products, the total effort is without value if the cooling portion of the process cannot provide supercooling prior to solidification that enables formation of the desired structure. Furthermore, the subsequent controlled cooling must be sufficiently uniform to avoid damaging thermal stresses. Present unknowns in this element of the process, therefore, are:

- o Cooling rates to achieve supercooling prior to solidification compatible with elimination of devitrification, or with formation of polycrystalline oxides with acceptable crystal size;
- o Effects of sample size on cooling rates, and vice versa;
- o Effects of cooling rates on induced thermal stress, and effects of those stresses on product properties.

III.6.1.2 Heating, Melting, and Positioning

Other critical steps in the selected processing approach are those which operate on the material prior to cooling - the heating and positioning steps.

While the zero "G" of orbital flight provides the levitation which enables crucibless processing, the small disturbances (solar panel rotation, crew movement, attitude control, etc.) which are part of such flight induce instabilities, or relative motion between the levitated specimen and the rest of the spacecraft. A positioning system is required, which counteracts these instabilities, which maintains the specimen in the optimum location for heating, and which may be used to insert the specimen or remove the processed sample.

A wide variety of forces may be useful for such positioning - gas streams, acoustic pressure, electromagnetic fluxes, and possibly, electrostatic charges. On the other hand, the interactions of the solid and molten transparent oxides with the methods which may be feasible for heating the specimens, with the chemistry of materials associated with various positioning methods, as well as with the thermodynamic processes of these methods, may be deleterious to the desired products. For potential heating methods such as hot wall, solar or arc imaging, electron beam, laser, and possibly induction (with a susceptor), difficulties arise in attempting to combine various positioning and heating methods. For instance, gas stream and acoustic pressure positioning require a gas surrounding the specimen, whereas electron beam heating requires a moderate vacuum. Further, hard vacuum is likely to induce evaporation and decomposition of the molten oxides under consideration, while gases could introduce contaminants and nucleation sites in the melt. Further discussion of these critical areas will be found in Appendix B, Book 2 of Volume II of this Report.

In summary, the knowledge "gaps" related to the critical process elements; positioning, heating and melting are:

- o Performance characteristics of various heating methods; solar and arc imaging, laser, heated walls, induction (with susceptor), electromagnetic (microwave cavity), electron beam. To include heating rates, temperature distribution and response, melting rates, etc. and their governing parameters.
- o Performance characteristics of various positioning methods; acoustic, gas stream, electrostatic, electromagnetic. To include controllable specimen size, positioning accuracy, controllable accelerations and velocities, etc., and their governing parameters.

III.6.1.3 Materials

As an initial effort in selecting from among the processing techniques discussed above, and to establish a baseline of raw materials for this process, it is

important to increase the presently documented data characterizing the available forms of the oxides under consideration. Such raw materials, available in various forms from various sources, will contain various amounts and types of impurities. The unknowns, then are:

- o For each source, the form of available oxides,
- o The types and amounts of impurities.

Closely related to the materials characterization, is the area of material response to processing conditions. Such information is important to establishing the thermal/atmosphere/duration guidelines for the various process steps. Temperature levels for degassing, degassing rates, melting, superheating, dwell times at superheat, phase change conditions, etc. must be determined. Thus, information is required on

- o Types and rates of gas contaminant efflux versus temperature,
- o Effects of various levels of vacuum and various inert gas atmospheres on contaminant efflux and level during degassing and melting.

III.6.1.4 Other Areas

The interactions among the various process steps and their combined effects when carried out in the selected sequence are, at present, unknown. Lack of experimental data has been supplanted by judgement during this Study, but the feasibility of the selected approach remains speculative until evaluation can be made of

- o Process development and design data for combined positioning, heating and cooling in the sequences defined for the selected approach

If all feasibility milestones are met successfully, the more standard engineering design and manufacturing problems will remain to be pursued. Typically, those efforts will seek information on

- Performance and design parameters of full scale equipment
- Loading and response of equipment during mission cycle
- Scale-up parameters

Finally, while not planned for initial phases of processing transparent oxides, there would appear to be considerable benefit from forming such materials into shapes (other than spheroids) from the melt. For instance, oblate spheroids would allow cutting of larger diameter lenses. Remelting of the space-produced product on the ground would, of course, return the material to an undesirable crystalline structure. If forming the shapes in space becomes of interest, then information on capabilities of the various forming methods would be required. These include mechanical, gas jets, electromagnetic, centrifugal, etc. Data would be required on

- Variety of achievable shapes versus forming method
- Accuracy of achievable shapes
- Effects of methods on product quality.

III.6.2 EXPERIMENT REQUIREMENTS FOR VERIFICATION OF SELECTED APPROACH FOR PROCESSING TRANSPARENT OXIDES

The preceding discussion, with its heavy emphasis on the lack of some very basic information, is indicative of the highly experimental nature of this area. Primarily, it is a question of whether the advantages of zero "G" (containerless melting and solidification, uniform surface cooling, and lack of convection in the melt) will overcome the devitrification that occurs when nucleation from these sources take place in ground-based processing.

It is felt (Section III.2) that the many years of analyses and the limited conditions available for experimentation in the area of transparent oxides have

left the current state-of-the-art in the position that only free suspension processing experiments and tests can provide the final answers as to the feasibility of this Idea. Consequently, the previously identified knowledge "gaps" have been utilized as a point of departure to define the requirements for such experiments and tests.

Since free suspension processing is still in its infancy for more common materials, and barely into the conceptual stage for the oxides under study here, early experiments are required simply to establish methods by which elements of the processing may be carried out.

Such early efforts are required to select, from among potential positioning and heating methods, the best combination compatible with the properties and behavior of the oxides under study.

In conjunction with that determination, it is also an early task to perform tests to characterize the available forms of these oxides so that their response to processing environments and their pre-processing characteristics are known.

Once the above information has been obtained, experiments and tests are required to assess the feasibility of the total process. Successful demonstration of process feasibility through flight test will be the basis for prognostication of a beneficial space processing application, and should lead to the design and development of a prototype system for orbital demonstration. Tests are likely to be required for the engineering development of parts and components of this prototype, which would subsequently be proof-tested on a Shuttle Sortie Lab flight.

Specific requirements for experiments and tests to carry out the program outlined above are summarized in Figures III-49 to III-57.

III.6.3 SUMMARY OF KNOWLEDGE GAPS, EXPERIMENT AND TEST REQUIREMENTS OF PROCESSING TRANSPARENT OXIDES

The overall relationships of the previously discussed knowledge gaps, required experiments and tests, and their requirements in shown in Figure III-58A and III-58B.

PURPOSE:

TO CHARACTERIZE VARIOUS SAMPLES OF ALUMINA, ZIRCONIA, YTTRIA. TO SELECT A SAMPLE PREPARATION METHOD (AND POSSIBLY A SUPPLIER) THAT WILL YIELD SAMPLES LIKELY TO FORM GLASSES WHEN MELTED IN LEVITATED CONDITION.

CONDITIONS:

STANDARD GROUND LABORATORY, CONTROLLED HEATING ENVIRONMENT TO 3000°C. INERT GAS ATMOSPHERES AT PRESSURES UP TO ONE ATM, DOWN TO VACUUM ($\sim 10^{-3}$ N/M^2). TEMPERATURE REGULATION TO CONTROL COOLING FROM $\sim$3000°C AT 10 TO 1000°C/SEC.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

ALUMINA, ZIRCONIA, YTTRIA RODS 1 CM DIAMETER, 3 TO 4 CM LONG, WEIGHT UP TO 16 GM PER SAMPLE, VARIOUS HIGH PURITIES PRODUCED BY VARIOUS SUPPLIERS, BY VARIOUS METHODS.

EQUIPMENT, APPARATUS REQUIRED:

FURNACE WITH CONTROLLED ATMOSPHERE TO $\sim$3000°C. MASS SPECTROMETER, TRACE X-RAY CRYSTALLOGRAPHY EQUIPMENT. POWER SUPPLY $\sim$3 KW, 60 HZ.

PROCEDURE:

ANALYZE SAMPLES FOR TRACE IMPURITIES (1 TO 2 DAYS). MELT SAMPLES (2 HOURS). X-RAY CRYSTALLOGRAPHY (1 DAY). CHEMICAL ANALYSIS OF SPECIMENS AFTER MELT IN VACUUM (1 DAY).

Figure III-49. Summary Definition of Requirements for Verification Experiments or Tests, I - Evaluation of Candidate Sample Materials

PROCESSING TRANSPARENT OXIDES

PURPOSE:

TO CHARACTERIZE DEGASSING REACTIONS, SUBLIMATION OF VOLATILE CONSTITUENTS, COMPOSITION, AND PHASE CHANGES WHICH ALUMINA, ZIRCONIA, AND YTTRIA MAY EXHIBIT UNDER CONDITIONS OF VACUUM AND UNDER INERT GAS ATMOSPHERE.

TO FORMULATE ENVIRONMENTAL CONDITIONS TO CONTROL THESE GAS-SOLID, GAS-LIQUID REACTIONS.

CONDITIONS:

AS IN I.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

10 GRAM CUBES OF ALUMINA, YTTRIA, ZIRCONIA PREPARED BY SINTERING OR BY SPECIAL TECHNIQUE SUCH AS CARBON IMAGING FURNACE.

EQUIPMENT, APPARATUS REQUIRED:

FURNACE SUCH AS THE CARBON ARC IMAGING FURNACE (FOCUSSED RADIATION) OR LASER OR ELECTRON BEAM FURNACE (VACUUM ONLY), MASS SPECTROMETER, RADIATION PYROMETER, VACUUM SYSTEM AND INERT GAS ADMISSION SYSTEM, MATERIALS CHARACTERIZATION AND EVALUATION EQUIPMENT, COMPUTER FOR DATA ANALYSIS.

PROCEDURE:

PREPARATION OF SPECIMEN (2 DAYS), LOAD FURNACE (MINUTES). INITIATE AND ADJUST HEATING, VACUUM, GAS ADMISSION (1/2 HOUR). HEAT AND MELT UNDER SPECIFIED ENVIRONMENTAL CONDITIONS, MONITOR GAS ENVIRONMENT AND TEMPERATURE (1 HOUR). ALLOW TO COOL (MINUTES). PERFORM MATERIALS CHARACTERIZATION AND EVALUATION (2 DAYS).

Figure III-50. Summary Definition of Requirements for Verification Experiments or Tests, II - Initial Processing Studies

PROCESSING TRANSPARENT OXIDES

PURPOSE:

EVALUATION AND SELECTION OF HEATING TECHNIQUE OR TECHNIQUES.
EVALUATION OF HEATING RESPONSES.

CONDITIONS:

AS IN I. WITH ADJUSTED RESIDUAL GAS SPECIES AND PARTIAL PRESSURES.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

10 TO 100 GRAM SPECIMENS OF YTTRIA, ALUMINA, ZIRCONIA.

EQUIPMENT, APPARATUS REQUIRED:

HEATING METHOD CANDIDATES: FOCUSSED RADIATION (SOLAR, CARBON ARC), LASER, HOT WALL, ELECTRON BEAM, ELECTROMAGNETIC. HEATING CHAMBER TO ACCOMMODATE EACH HEATING TECHNIQUE, VACUUM SYSTEM WITH GAS ADMISSION SYSTEM, MASS SPECTROMETER, RADIATION PYROMETER, MATERIALS EVALUATION AND CHARACTERIZATION EQUIPMENT, COMPUTER FOR DATA ANALYSIS, MELT WEIGHT, LENGTH MEASUREMENT AND TIMING APPARATUS.

PROCEDURE:

PLACE SPECIMEN IN HEATING UNIT (MINUTES). HEAT AND MELT, MONITORING RESUDUAL GAS SPECIES AND PARTIAL PRESSURES, TEMPERATURE, TIME, AMOUNT MELTED (UP TO 3 HOURS). COOL (UP TO 5 HOURS). MATERIALS CHARACTERIZATION AND EVALUATION (1 WEEK). TIME - TEMPERATURE - GAS DATA ANALYSIS (2 DAYS). REPEAT FOR EACH TECHNIQUE.

Figure III-51. Summary Definition of Requirements for Verification Experiments or Tests, III - Initial Heating and Melting Studies

PURPOSE:

EVALUATION AND SELECTION OF POSITION CONTROL TECHNIQUE FOR CONTAINERLESS PROCESSING. EVALUATION OF PERFORMANCE CHARACTERISTICS OF POSITION CONTROL SYSTEMS.

CONDITIONS:

INITIALLY, STANDARD LABORATORY ENVIRONMENT WITH SPECIMEN LEVITATION AND CONTROLLED VACUUM OR ATMOSPHERE, AS REQUIRED FOR PARTICULAR POSITION CONTROL TECHNIQUES. PROVISION FOR PREHEATING, WHERE REQUIRED, TO ~2000°C. LATER, ZERO "G" ENVIRONMENT FOR TESTING OF SELECTED TECHNIQUE(S). ALSO PROVISION FOR VACUUM OR ATMOSPHERE, AND PREHEATING, WHERE REQUIRED.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

MAXIMUM WEIGHT OF ZIRCONIA, YTTRIA, OR ALUMINA COMPATIBLE WITH POSITION CONTROL TECHNIQUE UNDER TEST. APPARATUS FOR PRODUCING AND POSITIONING FORCES; ACOUSTIC, GAS STREAM, ELECTROMAGNETIC.

EQUIPMENT, APPARATUS REQUIRED:

INITIALLY, GROUND LABORATORY SYSTEM. PREHEATING APPARATUS. INSTRUMENTATION FOR RECORDING, DETERMINING POSITIONING VELOCITIES, ACCELERATIONS, FORCES AND STABILITY, ENERGY USAGE.

LATER, DROP TOWER, KC-135, SOUNDING ROCKET FOR ZERO "G" EVALUATION OF SELECTED TECHNIQUE(S). COMPUTER FOR ANALYSIS OF POSITION, VELOCITY, AND ACCELERATION VERSUS TIME DATA.

PROCEDURE:

FOR GROUND-BASED TESTS, PREHEAT WHERE REQUIRED (~MINUTES). LEVITATE AND STABILIZE SPECIMEN UNDER VARIOUS INITIAL DISTURBANCE CONDITIONS, VARIOUS LEVELS OF POSITIONING FORCE (~1 HOUR).

FOR DROP TOWER TESTS, PERFORM DROP TOWER PACKAGE INSTALLATION (~1 DAY). PERFORM DROP PROCEDURE (3 TO 10 SECONDS). REPEAT FOR EACH SELECTED POSITIONING TECHNIQUE.

FOR KC-135, INSTALL KC-135 PACKAGE (~2 HOURS). PERFORM FLIGHT PROCEDURE; TAKE-OFF, CLIMB, DIVE, PULL-UP, KEPPLERIAN TRAJECTORY (20 TO 40 SECONDS), PULL-OUT.

Figure III-52. Summary Definition of Requirements for Verification Experiments or Tests, IV - Initial Levitation and Positioning Studies (IV in Ground Laboratories, IVA on Drop Tower, KC-135 Sounding Rocket)

PURPOSE:

TO PROVIDE INITIAL EVALUATION OF SELECTED PROCESS. TO OBTAIN PROCESS DEVELOPMENT DATA AND ESTABLISH CONCEPTUAL HARDWARE DESIGN FOR ZERO "G" FLIGHTS.

CONDITIONS:

AS IN IV, VACUUM OR INERT GAS WITH RESIDUAL GAS SPECIES AND PARTIAL PRESSURES ADJUSTED FOR PROCESSING, SPECIMEN LEVITATED AGAINST ONE "G" ENVIRONMENT, CONTROLLED TEMPERATURE PROFILES.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

10 TO 100 GRAM SPECIMENS OF YTTRIA, ALUMINA, ZIRCONIA. EQUIPMENT REPRESENTING SELECTED TECHNIQUES FOR POSITIONING, HEATING, MELTING, COOLING.

EQUIPMENT, APPARATUS REQUIRED:

PROCESSING CHAMBER, CONTROLLED GAS ENVIRONMENT, LEVITATION UNIT, MASS SPECTROMETER, RADIATION PYROMETER, MATERIALS CHARACTERIZATION AND EVALUATION APPARATUS, AND COMPUTER FOR DATA ANALYSIS.

PROCEDURE:

SPECIMEN PREPARATION (2 DAYS). PRE-TEST MATERIALS EVALUATION AND CHARACTERIZATION (1 WEEK). LOAD SPECIMEN INTO CHAMBER, SYSTEM INTEGRATION AND WARM UP PROCEDURE (2 HOURS). LEVITATION, HEATING, AND MELTING (2 HOURS). SUPERHEATING (10 MINUTES). SUPERCOOLING AND COOLING ALONG SELECTED THERMAL PROFILE (3 TO 5 HOURS). POST-TEST MATERIALS EVALUATION AND CHARACTERIZATION (1 WEEK).

Figure III-53. Summary Definition of Requirements for Verification Experiments or Tests, V - Levitation, Positioning, Heating, Melting, Cooling, Yttria, Zirconia, and Alumina

PURPOSE:

TO PROVIDE INITIAL ZERO "G" EVALUATION OF SELECTED PROCESS. TO OBTAIN PROCESS DEVELOPMENT DATA AND REFINE HARDWARE DESIGN.

CONDITIONS:

ZERO "G", AS IN IVA.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

10 TO 100 GRAMS YTTRIA, ZIRCONIA, ALUMINA. POSITIONING APPARATUS, HEATING AND MELTING UNIT, COOLING SYSTEM, REPRESENTATIVE OF SELECTED TECHNIQUES.

EQUIPMENT, APPARATUS REQUIRED:

SOUNDING ROCKET, PHOTOGRAPHIC INSTRUMENTATION, GAS SAMPLE COLLECTION EQUIPMENT, RADIATION PYROMETER, OTHER PROCESS INSTRUMENTATION, QUENCHING UNIT FOR COLLECTION OF SPECIMEN, RECOVERY PACKAGE, MATERIALS EVALUATION AND CHARACTERIZATION LAB, COMPUTER FOR DATA ANALYSIS.

PROCEDURE:

EXPERIMENT PACKAGE PRE-FLIGHT PREPARATION (1 WEEK). PRE-LAUNCH PROCESSING (1 WEEK). LAUNCH (MINUTES). ZERO "G" HEATING, MELTING AND COOLING DURING FLIGHT WITH POSITION CONTROL AND GAS-TEMPERATURE-TIME PROFILE MONITORING. QUENCH AT END OF WEIGHTLESS FLIGHT, IF DESIRED (6 TO 10 MINUTES). RETURN OF SAMPLES (1 TO 2 DAYS). MATERIALS CHARACTERIZATION AND EVALUATION (1 WEEK).

Figure III-54. Summary Definition of Requirements for Verification Test, VA - Zero "G" Positioning, Heating, Melting, and Cooling of Oxides

PURPOSE:

TO EVALUATE TECHNIQUES FOR SHAPING TRANSPARENT OXIDES INTO DESIRABLE CONFIGURATIONS DURING COOLING FROM THE MELT.

CONDITIONS:

INITIALLY, STANDARD GROUND LABORATORY AS IN V. LATER, ZERO "G" SHUTTLE SORTIE LABORATORY ENVIRONMENT. BOTH WITH SPECIMEN PREHEATING TO 2000°C (IF REQUIRED), CONTROLLED (VACUUM OR INERT GAS) ATMOSPHERE AND HEATING TO 3000°C IN SELECTED TEMPERATURE PROFILE, COOLING IN SELECTED PROFILE (QUENCHING, IF REQUIRED) TO HANDLING TEMPERATURE (~100°C).

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

10 TO 100 GRAM SPECIMENS OF YTTRIA, ZIRCONIA, ALUMINA. VARIOUS DEVICES FOR FORMING SHAPES FROM THE MELT BY MECHANICAL WORKING, GAS JETS, ELECTROMAGNETIC FORCES, SPINNING, FREE CASTING, ETC. SORTIE LAB TESTS TO INCLUDE ONLY THE MORE PROMISING TECHNIQUES FROM GROUND LAB EXPERIMENTS.

EQUIPMENT, APPARATUS REQUIRED:

GROUND LABORATORY: CHAMBER WITH APPARATUS FOR SPECIMEN PREHEATING, LEVITATION, POSITIONING, HEATING, SUPERHEATING, COOLING, SUPERCOOLING, QUENCHING. INSTRUMENTATION, PHOTOGRAPHY FOR RECORDING FORMING PROCESS AND RESULTS.

SHUTTLE SORTIE LABORATORY: FREE SUSPENSION PROCESSING SYSTEM WITH ABOVE CAPABILITIES AND NO LEVITATION SYSTEM. INSTRUMENTATION AND RECORDING AS ABOVE. X-RAY CRYSTALLOGRAPHY EQUIPMENT.

PROCEDURE:

GROUND LABORATORY: INSERT SPECIMEN IN CHAMBER (SECONDS), PREHEAT TO ~2000°C (~MINUTES), LEVITATE AND STABILIZE SPECIMEN (~MINUTE), HEAT AND MELT PER SELECTED PROFILE (2 HOURS, INITIATE COOLING AND SUPERCOOLING (5 TO 7 HOURS), EXERCISE FORMING TECHNIQUE DURING COOLING AND SUPERCOOLING, MEASURE AND ANALYZE RESULTING PRODUCT (1 WEEK). REPEAT FOR EACH FORMING METHOD.

SORTIE LABORATORY: AS ABOVE, EXCEPT NO LEVITATION OF SPECIMEN. REPEAT FOR EACH SELECTED TECHNIQUE AND MATERIAL SPECIMEN.

Figure III-55. Summary Definition of Requirements for Verification Experiments or Tests, VI - Techniques for Zero "G" Forming of Transparent Oxides (VI in Ground Laboratory, VIA on Shuttle Sortie)

PURPOSE:

TO OBTAIN ENGINEERING DESIGN DATA, TO ADVANCE DEVELOPMENT OF PROCESSING EQUIPMENT, TO VERIFY PERFORMANCE OF EQUIPMENT, TO QUALIFY EQUIPMENT FOR FLIGHT.

CONDITIONS:

GROUND LABS: VARIOUS ENVIRONMENTS FROM ROOM CONDITIONS TO SIMULATED SORTIE LAB CONDITIONS, DEPENDING ITEM BEING TESTED. TEMPERATURES TO $\backsim 3000^{\circ}$ FOR COMPONENTS TO BE EXPOSED TO MELT. VACUUM TO 10^{-3} N/M^2 FOR COMPONENTS EXPOSED TO PROCESSING VACUUM (IF REQUIRED).

SORTIE LAB OR PALLET: NOMINAL FLIGHT CONDITIONS.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

PARTS, COMPONENTS, UNITS OF TRANSPARENT OXIDE PROCESSING SYSTEM (IN VARIOUS STAGES OF DEVELOPMENT).

EQUIPMENT, APPARATUS REQUIRED:

STANDARD DEVELOPMENT LABORATORIES (ELECTRICAL, ELECTRONIC, MECHANICAL, ETC. SHAKE TABLES, THERMAL AND VACUUM CHAMBERS, CENTRIFUGE, ANECHOIC CHAMBER. SORTIE LAB WITH POWER SUPPLY, THERMAL CONTROL SYSTEM, TEST MONITOR AND CONTROL CONSOLE. EQUIPMENT INSTRUMENTATION, PHOTOGRAPHY, DATA RECORDING AND PROCESSING.

PROCEDURE:

VARIES WITH EQUIPMENT UNDER TEST. EQUIPMENT INSTALLATION, CHECKOUT, INITIATE INSTRUMENTATION AND RECORDING TEST AT LOW LOAD, FULL LOAD, QUALIFYING LOAD. MONITOR RESPONSES AND PERFORMANCE, PERFORM NECESSARY MODIFICATIONS, MAINTENANCE, REPAIR. (1/2 HOUR TO 5 DAYS PER TEST).

Figure III-56. Summary Definition of Requirements for Development Tests, VII - Transparent Oxide Processing Equipment Design Data, Development, Qualification (VII in Ground Laboratories, VIIA on Sortie Lab or Pallet)

PURPOSE:

TO ESTABLISH THE CAPABILITIES AND LIMITATIONS OF THE TRANSPARENT OXIDE PROCESSING SYSTEM AS REGARDS FULL-SCALE OPERATIONS (PRODUCT QUALITY, QUANTITY, TIMING).

CONDITIONS:

SORTIE LAB ENVIRONMENT.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

PROTOTYPE TRANSPARENT OXIDE PROCESSING SYSTEM (INCLUDING ALL AUTOMATED AND MANUAL STEPS, NOMINAL INSTRUMENTATION AND CONTROLS. RAW MATERIALS FOR 5 DAY PRODUCTION RUN. NOMINAL MAINTENANCE AND REPAIR KIT.

EQUIPMENT, APPARATUS REQUIRED:

TEST INSTRUMENTATION, PHOTOGRAPHIC AND DATA RECORDING. NOMINAL POWER AND THERMAL CONTROL SUPPORT, COMMUNICATIONS, DISPLAYS AND CONTROLS.

PROCEDURE:

LOAD RAW MATERIALS. INITIATE PROCESSING. PERFORM MANUAL OPERATIONS (WHERE REQUIRED). PERFORM MONITORING AND CONTROL. RECORD PROCESS PARAMETERS AND TEST DATA. PERFORM MAINTENANCE AND REPAIR, AS REQUIRED. PACKAGE PRODUCTS FOR RETURN. SHUT DOWN SYSTEM, AND STOW (AS REQUIRED).

Figure III-57. Summary Definition of Requirements for Development Test, VIII - Prototype/Proof Test

KNOWLEDGE GAPS	EXPERIMENTS AND VERIFICATION TESTS	EXPERIMENT AND TEST REQUIREMENTS
① EVALUATION OF SPECIFIC OXIDES	PURITY ANALYSIS, MELTING, X-RAY EXAMINATION, ALUMINA, ZIRCONIA, YTTRIA, FROM VARIOUS SOURCES, PRODUCED BY VARIOUS PROCESSES. SMALL SAMPLES, HIGH PURITY, IN HARD VACUUM AND INERT GAS. TO SELECT INITIAL MATERIALS MOST LIKELY TO PRODUCE DESIRED GLASSES VIA SPACE PROCESSING.	STANDARD GROUND LAB WITH CONTROLLED ATMOSPHERE FURNACE (~ 3000°C), SPECIMENS OF HIGH PURITY ALUMINA, ZIRCONIA, YTTRIA FROM VARIOUS SOURCES, TRACE IMPURITY ANALYSIS EQUIPMENT, X-RAY CRYSTALOGRAPHY, VACUUM SYSTEM, INERT GAS SYSTEM, 1-2 DAY PER SPECIMEN. MANUAL
② DEFINITION OF PROCESSING CONDITIONS	HEATING, MELTING SELECTED OXIDES TO DETERMINE EFFECTS OF VACUUM LEVEL AND/OR INERT GAS ATMOSPHERE, NEED FOR CONTAMINANT REMOVAL. ALSO DEGASSING REACTIONS, PHASE CHANGES.	STANDARD GROUND LAB WITH FURNACE(S) ACCOMMODATING CARBON ARC-IMAGING HEATER OR LASER, AND ELECTRON BEAM GUN, VACUUM AND INERT GAS. SELECTED SPECIMENS OF ALUMINA, ZIRCONIA, YTTRIA. MASS SPECTROMETER, RADIATION PYROMETER, VACUUM SYSTEM AND INERT GAS SYSTEM. MATERIALS CHARACTERIZATION LAB. 4 HOURS PER SPECIMEN RUN, 2 DAYS PER SPECIMEN FOR CHARACTERIZATION. MANUAL
③ MATERIAL HEATING METHODS AND RESPONSES	HEATING EXPERIMENTS WITH SOLAR ARC, LASER, HEATED CHAMBER, ELECTROMAGNETIC, ELECTRON BEAM EQUIPMENT ON SELECTED OXIDES. TO ESTABLISH TIME, TEMPERATURE RESPONSE, MELTING RATE, EFFECTS ON IMPURITIES, UNIFORMITY OF HEATING, OTHER EFFECTS, AND SELECT MOST EFFICIENT, COMPATIBLE HEATING METHOD. INVESTIGATE COOLING EFFECTS.	STANDARD LAB AS ABOVE PLUS ACCOMMODATION FOR SOLAR IMAGING, HOT CHAMBER AND ELECTROMAGNETIC HEATING. OTHER REQUIREMENTS AS ABOVE PLUS MELT-MEASURING APPARATUS AND COOLING EQUIPMENT. 8 HOURS PER SPECIMEN RUN, 2 DAYS FOR CHARACTERIZATION.
④ MATERIAL POSITIONING METHODS AND RESPONSES	EXPERIMENTS ON POSITIONING CAPABILITY OF ACOUSTIC, GAS STREAM, ELECTROSTATIC FORCES VS. SOLID ALUMINA, ZIRCONIA, YTTRIA; (MICROWAVE FORCES AS BACKUP). TO ESTABLISH MAXIMUM CONTROLLABLE SPECIMEN MASS, POSITION, VELOCITY, ACCELERATION LEVELS, AND TO SELECT MOST EFFECTIVE, COMPATIBLE CONTROL METHOD.	STANDARD LAB WITH LEVITATION APPARATUS. VARIOUS WEIGHTS OF SPECIMENS. ACCOMMODATION FOR ACOUSTIC, GAS STREAM, ELECTROSTATIC, MICROWAVE POSITIONING DEVICES. POSITION SENSING, VELOCITY AND ACCELERATION MEASURING EQUIPMENT, PHOTOGRAPHY. AUTOMATED POSITIONING, REMAINDER MANUAL. LATER, DROP TOWER, KC-135 AND SOUNDING ROCKET. SELECTED POSITIONING METHOD(S) FROM ABOVE. LARGEST SPECIMEN WEIGHTS COMPATIBLE WITH TEST PACKAGE. INTEGRATED PACKAGE OF POSITIONING DEVICE, INSTRUMENTATION, POWER SUPPLY, DATA RECORDING, TELEMETRY. AUTOMATED-DURATION OF RUNS-DROP TOWER, 3-10 SEC; SOUNDING ROCKET, 6-10 MIN. TELEMETRY OR RECORDING OF DATA, RECOVERY OF TEST PACKAGE.

Figure III-58A. Definition of Requirements for Experiments to Verify Selected Approach For Transparent Oxide Processing

KNOWLEDGE GAPS	EXPERIMENTS AND VERIFICATION TESTS	EXPERIMENT AND TEST REQUIREMENTS
(5) PROCESS FEASIBILITY	TEST OF COMBINED POSITIONING, HEATING, MELTING, COOLING UNDER SELECTED CONDITIONS TO MAKE PRELIMINARY PROCESS EVALUATION, ACCRUE DEVELOPMENT AND DESIGN DATA.	STANDARD LAB AS IN (2) (3) (4) ABOVE UNDER CONDITIONS, AND WITH APPARATUS, SELECTED AS RESULT OF (2) (3) (4). PROVISION FOR MODIFICATION OF CONDITIONS, APPARATUS TO OPTIMIZE PERFORMANCE. AUTOMATED WITH PROVISION FOR MANUAL OVERRIDE. 7 TO 9 HOURS PER RUN. LATER, SOUNDING ROCKET WITH TEST PACKAGE BASED ON DESIGN DATA AND CONDITIONS DERIVED FROM GROUND LAB TESTS. TEST PACKAGE TO INCLUDE APPARATUS FOR POSITIONING, HEATING, MELTING, COOLING PER PREPROGRAMMED PROFILE AND SEQUENCE, POWER SUPPLY, INSTRUMENTATION, DATA RECORDING, TELEMETRY OF DATA, RECOVERY OF TEST PACKAGE. 6–10 MINUTES. AUTOMATED.
(6) FORMING TECHNIQUES (FUTURE POSSIBILITY)	EXPERIMENTS ON ACHIEVABLE SHAPE VARIETY, ACCURACY, OF ELECTROSTATIC, OTHER TECHNIQUES.	STANDARD LAB AND EQUIPMENT OPTIMIZED FROM (5). ACCOMMODATING APPARATUS FOR SEVERAL FORMING TECHNIQUES (E.G., GAS JETS, ELECTROMAGNETIC) INCLUDING CENTRIFUGAL CASTING WITH AND WITHOUT MOLD), GAS DYNAMIC, ETC. MANUAL. 1 TO 2 HOURS PER RUN. LATER, SHUTTLE SORTIE LAB, APPARATUS FOR SELECTED FORMING TECHNIQUES, OTHER REQUIREMENTS AS IN SHUTTLE TEST OF (7). 7 TO 9 HOURS PER RUN.
(7) EQUIPMENT DESIGN DATA	DEVELOPMENT, ENGINEERING, QUALIFICATION TESTS.	STANDARD LAB WITH SHAKE TABLES, VACUUM AND THERMAL CHAMBERS, CENTRIFUGE, ETC. TESTING TO CONFIRM ADEQUACY OF DESIGN FOR SHUTTLE SORTIE LOADS AND CONDITIONS, FOR COMPATIBILITY AMONG PROCESSING EQUIPMENTS, FOR ESTIMATING/CONFIRMING PERFORMANCE OF EQUIPMENTS, ETC. INSTRUMENTATION FOR LOADING, ENVIRONMENT, RESPONSE, RECORDING OF DATA, PHOTOGRAPHY. LATER SHUTTLE SORTIE LAB AND/OR PALLET WITH ACCOMMODATION FOR COMPONENTS, SUBSYSTEMS OF PROCESSING SYSTEM. SUPPORT UTILITIES AND INSTRUMENTATION TO VERIFY DESIGN PERFORMANCE AND RESPONSE. TELEMETRY OF DATA, RECOVERY OF TESTED EQUIPMENT. 1/2 HOUR TO 5 DAYS PER TEST.
(3) PRODUCTION DATA	PROTOTYPE TEST FOR PRODUCT UNIFORMITY, QUANTITY.	SHUTTLE SORTIE LAB WITH PROTOTYPE TRANSPARENT OXIDE PROCESSING SYSTEM AND ESTIMATED 5 DAY PRODUCTION AMOUNT OF SELECTED OXIDE (S). PERFORMANCE AND LOADS INSTRUMENTATION. PRODUCTION-TYPE AUTOMATION, OPERATOR DISPLAYS AND CONTROLS. TELEMETRY OF DATA, RECOVERY OF PRODUCTS AND PROCESSING SYSTEM. 5 DAY DURATION.

Figure III-58B. Definition of Requirements for Experiments to Verify Selected Approach For Transparent Oxide Processing

III.7 DEFINITION OF REQUIREMENTS FOR EXPERIMENTS TO VERIFY SELECTED APPROACH FOR FABRICATING HIGH PURITY TUNGSTEN X-RAY TARGETS

III.7.1 CRITICAL ELEMENTS IN SELECTED APPROACH FOR FABRICATING HIGH PURITY TUNGSTEN X-RAY TARGETS, AND RELATED KNOWLEDGE "GAPS"

Considerable experimental effort has already been carried out in the search for materials and ground processes which could produce x-ray targets with the required room-temperature ductility. Figure III-19, shown earlier, is a small sample of the variety of approaches that have been tested.

Levitation melting (and solidification) of tungsten in ground facilities is presently under active study, and promises to provide some relief from the problem of contamination of the melt by the crucible. Controlled cooling of the melt, however, will still remain a problem in earth-bound processing since the electromagnetic forces required to levitate the tungsten also cause heating. Furthermore, ground-based levitation cannot eliminate heat-induced convection in the melt.

Processing of tungsten in the zero "G" of orbital flight, selected as the best approach in Section III.3, appears to offer the possibility of overcoming both of these problems.

Within that selected approach, however, there are several critical steps which involve technical areas where sufficient knowledge is presently lacking. These steps and their associated knowledge "gaps" are discussed below.

III.7.1.1 Degassing of the Raw Material

Degassing of the tungsten is required to remove the interstitial impurities which cause embrittlement, and lead to a high ductile-brittle transition temperature. While such a process step is expected to benefit from heating in a hard vacuum such as that available in orbit, certain embrittlement-causing elements may require

more complex treatment. Carbon, for instance, will require a partial pressure of oxygen to enable the decarburization of the tungsten.

Degassing also serves to minimize the occurence of bubbles in the melt which lead to porosity and voids in the solidified tungsten.

An alternative degassing approach, heating in an inert gas, while not selected as the best approach, offers some possible advantages. Higher partial pressures of a pure inert gas, while slowing the diffusion of degassing reactants and products, inhibit bubble formation and tungsten evaporation.

In addition, hydrogen, introduced in the degassing step, could aid in removing sulphur and phosphorus from the tungsten.

Current state-of-art in assessing the achievable purification of tungsten by degassing is characterized by the limited information available on the following factors:

- o The influence of initial tungsten purity.
- o The residual gases in the tungsten, and their partial pressures at various degassing temperatures.
- o The optimum temperature(s) at which degassing should be performed.
- o The specific degassing chemical reactions which can take place in potential degassing environments.
- o The optimum duration (dwell time) of degassing step(s).
- o Tungsten vapor pressure
- o The best combination of solid-state and molten-state degassing durations, temperatures, and environments.

The key unknowns, of course, are the effects of these factors on the final tungsten properties, such as the amount of residual interstitial materials, grain size and shape.

III.7.1.2 Heating and Melting

Heating the tungsten to, and through, melt temperature without a crucible is the key step of this process. Following the solid phase degassing step, it is necessary to bring the total batch of tungsten to the completely molten state, so that subsequent solidification can take place in the absence of sites for heterogeneous nucleation.

Since little previous work has been reported on tungsten in the range of temperatures near, at, and above melting, characterization of that element in those ranges will be required.

A rapid heating cycle through, and above, melt temperature, has been incorporated in the selected approach in consideration of the amount of power required to maintain the required heat input to the tungsten at and above the melt temperature ($3410^{o}C$). Dwell time at the above-melt temperature, to evaporate such impurities may, in addition to imposing severe energy requirements (due to high radiation loss from the 3410+^{o}C tungsten), result in loss of tungsten by evaporation. The key consideration will be the vapor pressure of the tungsten as compared to the vapor pressure of the evaporating impurities. Such information, evaluated as a function of duration of dwell time, and compared to results obtained by heating and molten dwell in an inert gass-pressurized environment, are required for finalization of process steps.

There is a dearth of information on methods by which the tungsten can be heated in a crucibless process. Initial studies have led to the selection of radio frequency (RF)-induced eddy currents. While ground-based experiments on RF heating have been performed on low resistivity, low temperature, low density

materials such as aluminum, and analyses have been carried out on tungsten, new and verifying data are required, both on pertinent tungsten properties and on design and performance of RF-generating coil systems. Since RF has also been selected to provide positioning control for the tungsten during the space-based process steps, the required data must also account for that function.

The final item of interest in the heating and melting of tungsten is that of supplementary or alternative heating systems. Solar concentrators and electron beam guns have been considered, with present preference for electron beam, since it imposes no constraints on facility orientation, and could likely have other applications.

In summary, additional knowledge for heating and melting of tungsten is required in the following areas:

- o Characterization of tungsten - near, at, and above melt temperature (emissivity, vapor pressure, etc. of molten tungsten).
- o Heating and melting characteristics and effects:
  - The optimum dwell temperature(s).
  - The optimum dwell durations at and above melt temperature.
  - The most effective environments (vacuum, inert gas) during dwell.
  - The effects of initial tungsten purity.
- o Heating and melting equipment selection and design data:
  - Optimum RF coil shapes and sizes. } Integrated with
  - Heating and melting power requirements. } positioning
  - Evaluation of electron beam heating performance } requirements.

As noted earlier, the final measure of selected process variables resulting from acquiring data in the above areas will be the quality of the final tungsten product.

with intermittent injection of oxygen and/or hydrogen as required. Study of inert gas (10^5 N/M^2), however, provides a back-up in the event evaporation rates for tungsten in hard vacuum are excessive.

Degassing conditions are varied in early ground tests to establish material limits and constraints, and to provide early inputs to the processing system design. Subsequently, tests are required to establish the more rigorous requirements for positioning, heating (including auxiliary heating), and their integrated performance.

As data is acquired, and analyses confirmed (or alternatives indicated) more complex testing and more sophisticated facilities are employed.

When combined positioning and degassing techniques have been proven, the experiment program moves to the added complexity of melting and cooling.

Zero "G" testing (in drop towers, zero "G" aircraft, sounding rockets) is employed at various stages of the program to verify individual process steps or combinations of steps under conditions approaching those of orbital flight.

Finally, process equipment design data are verified in Shuttle flights of development equipment prior to prototype processing system test.

Requirements of specific experiments and tests to carry out the above program are summarized in Figures III-59 to III-68. In addition, Figure III-69 summarizes the requirements for operational testing of tungsten materials. This test is a ground test of tungsten samples produced in other experiments and tests. It is a standard method by which the User subjects new target materials to accelerated life testing in order to determine whether an improved material has been produced.

PURPOSE:

TO OBTAIN INITIAL DATA ON DEGASSING TIMES AND TEMPERATURE IN RELATION TO TYPE AND QUANTITY OF GASES AND/OR ELEMENTS (CARBON, ETC.) REMOVED. TO OBTAIN INITIAL DATA ON EFFECTS OF VACUUM DEGASSING ON MECHANICAL PROPERTIES OF TUNGSTEN, CHANGE IN INTERSTITIAL IMPURITY CONTENT, AND GRAIN SIZE AND SHAPE. TO AID IN SELECTION OF INITIAL TUNGSTEN PURITY.

CONDITIONS:

GROUND LABORATORY ENVIRONMENT WITH EXPERIMENT CONDITIONS OF 10^{-3} TO 10^{-6} N/M^2 VACUUM, 1800°C TO 2400°C.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

0.5 TO 10 GRAM SAMPLES OF COMMERCIAL AND HIGHER PURITY TUNGSTEN, 0.25 CM TO 2.7 CM CUBES

EQUIPMENT, APPARATUS REQUIRED:

CERAMIC OR TUNGSTEN SPECIMEN STING, VACUUM SYSTEM, SMALL VACUUM INDUCTION FURNACE, R.F. GENERATOR TO SUPPLY HEATING POWER TO VACUUM INDUCTION FURNACE, MASS SPECTROMETER AND GAS SAMPLING SYSTEM, TEMPERATURE MEASURING EQUIPMENT, MATERIALS CHARACTERIZATION AND EVALUATION EQUIPMENT, VACUUM FUSION GAS ANALYSIS EQUIPMENT, EMISSION SPECTROSCOPE OR SPARK GAP MASS SPECTROMETER, COMPUTER FOR DATA ANALYSIS AND EVALUATION.

PROCEDURE:

PRE-EXAMINATION, CHARACTERIZATION, AND EVALUATION OF SPECIMEN MATERIAL (1 WEEK). PREPARE SPECIMENS - INSPECT AND ANALYZE SAMPLE SPECIMENS AFTER PREPARATION (2 DAYS PER SPECIMEN). SET UP MASS SPECTROMETER AND GAS SAMPLING SYSTEM, HEATING, AND FIXTURING FOR VACUUM INDUCTION FURNACE EXPERIMENTS (1 HOUR). PERFORM HEATING IN VACUUM, DWELL 1 TO 20 MINUTES AT VARIOUS TEMPERATURES, MONITOR TIME, TEMPERATURE, PARTIAL PRESSURES, AND IDENTITY OF GASEOUS SPECIES, COOL IN VACUUM TO ROOM TEMPERATURE (3½ HOURS). INTERPRETATION, ANALYSIS, AND EVALUATION OF EXPERIMENTAL DATA USING COMPUTER (2 DAYS). POST-EXAMINATION, CHARACTERIZATION, AND EVALUATION OF SPECIMENS (1 WEEK). REPEAT 2 TO 4 TIMES FOR EACH SPECIMEN.

Figure III-59. Summary Definition of Requirements for Verification Experiments or Tests, 1 - Initial Vacuum Degassing Studies

PURPOSE:

TO OBTAIN DATA ON EQUIPMENT REQUIRED TO LEVITATE, POSITION AND CONTROL SOLID TUNGSTEN IN AN R.F. COIL.

CONDITIONS:

GROUND LABORATORY EQUIPMENT, VACUUM AT SAMPLE 10^{-3} TO 10^{-6} N/M^2 HEATING OF SAMPLE TO 1800°C TO 2400°C.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

10 TO 100 GRAM SAMPLES OF COMMERCIAL AND HIGH PURITY TUNGSTEN, 1 CM DIAMETER SPHERE OR EQUIVALENT ROD - APPROXIMATELY 1 CM DIAMETER X 1 CM LONG. SAMPLE WEIGHT TO VARY TO DETERMINE RF COIL DESIGN AND POWER REQUIREMENTS

EQUIPMENT, APPARATUS REQUIRED:

RF GENERATOR, LEVITATION/POSITIONING COIL, ELECTRONICS FOR MONITORING, POSITIONING AND STABILIZING SAMPLE, SAMPLE HEATING EQUIPMENT, OPTICAL PYROMETER, AND VACUUM SYSTEM.

PROCEDURE:

PREPARE SPECIMENS TO SHAPE (1 DAY). SET UP APPARATUS AND ELECTRONIC GEAR CONTROLS (1 HOUR). LEVITATE, HEAT (2½ HOURS). REPEAT FOR EACH SPECIMEN.

Figure III-60. Summary Definition of Requirements for Verification Experiments or Tests, 2 - Levitation of Solid Tungsten, Initial Studies

FABRICATION OF TUNGSTEN X-RAY TARGETS

PURPOSE:

TO DETERMINE ELECTRON BEAM HEATING AS A SUPPLEMENTARY SOURCE (TO RF) METHOD OF HEATING, MELTING AND SUPERHEATING TUNGSTEN.

CONDITIONS:

GROUND LABORATORY ENVIRONMENT WITH TEST CONDITIONS OF 10^{-3} TO 10^{-6} N/M^2, ELECTRON BEAM VOLTAGE BELOW 10 KV (OR X-RAY PROTECTION IS REQUIRED), SOLID STATE DEGASSING BEFORE MELTING OR USE DEGASSED SPECIMEN.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

10 TO 100 GRAM SAMPLES OF COMMERCIAL AND HIGH PURITY TUNGSTEN, ROD OR SPHERE 1 CM X 1 CM, ETC.

EQUIPMENT, APPARATUS REQUIRED:

VACUUM SYSTEM, POWER SUPPLY - 10 KV - 1 TO 2 AMPS, ELECTRON BEAM GUN, FIXTURING-TUNGSTEN STING ON NON-CONDUCTING SUPPORT OR RF LEVITATION TO HOLD MOLTEN TUNGSTEN, MASS SPECTROMETER FOR GAS ANALYSIS, TEMPERATURE MEASURING EQUIPMENT, PRE- AND POST-TEST MATERIALS CHARACTERIZATION AND EVALUATION EQUIPMENT, X-RAY SHIELDING.

PROCEDURE:

PREPARE SPECIMEN - INSPECT AND ANALYZE SAME MATERIAL (1 WEEK). MOUNT SPECIMEN IN VACUUM CHAMBER (2 HOURS). FOCUS ELECTRON BEAM GUN (10 MINUTES). PHYSICALLY SUSPEND OR LEVITATE SAMPLE (0 TO 20 MINUTES). HEAT SAMPLE (2 TO 3 HOURS). MEASURE TEMPERATURE AND POWER INPUT DURING TEST. MELT (10 MINUTES). COOL IN VACUUM (6 TO 20 MINUTES).

Figure III-61. Summary Definition of Requirements for Verification Experiments or Tests, 3 - Electron Beam Heating, Initial Studies

PURPOSE:

TO DETERMINE FEASIBILITY OF RF COIL POSITIONING WITH RF AND WITH ELECTRON BEAM HEATING FOR VACUUM DEGASSING. TO IDENTIFY TYPE AND AMOUNT OF GASES AND/OR ELEMENTS (CARBON, ETC.) REMOVED. TO EVALUATE RF INDUCTION HEATING AND ELECTRON BEAM HEATING OF LEVITATED SPECIMENS BY SPECIMEN PROPERTY DETERMINATIONS. TO ASSESS TEMPERATURE CONTROL OF LEVITATED SPECIMENS WITH LONG DURATION DWELLS.

CONDITIONS:

GROUND LABORATORY ENVIRONMENT WITH 10^{-3} TO 10^{-6} N/M^2 VACUUM AND 1800°C TO 2400°C AT SPECIMEN, SPECIMEN LEVITATION VIA RF FIELD, DWELLS AT SELECTED TEMPERATURES IN DEGASSING TEMPERATURE RANGE (1800°C TO 2400°C) WITH DWELL TIMES AS ESTABLISHED BY EXPERIMENT 1.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

10 GRAMS OR MORE (CAPABILITY ESTABLISHED BY EXPERIMENT) SPECIMENS OF COMMERCIAL AND HIGHER PURITY TUNGSTEN. 1 CM^3 OR MORE (CAPABILITY ESTABLISHED BY EXPERIMENT).

EQUIPMENT, APPARATUS REQUIRED:

VACUUM SYSTEM, LEVITATION CHAMBER, RF LEVITATION COIL MOUNTED IN CHAMBER, ELECTRON BEAM HEATING UNIT MOUNTED ON CHAMBER, FIXTURE FOR SAMPLE INSERTION INTO COIL IN VACUUM, RF POWER SUPPLY, (4 KW 450 KHZ TO 25 KW 450 KHZ,) MASS SPECTROMETER AND GAS SAMPLING SYSTEM, TEMPERATURE MEASURING INSTRUMENTATION, HYDROGEN GAS SYSTEM, MATERIALS CHARACTERIZATION AND EVALUATION APPARATUS, VACUUM FUSION GAS ANALYSIS EQUIPMENT, EMISSION SPECTROSCOPE OR SPARK GAP MASS SPECTROMETER, COMPUTER FOR DATA ANALYSIS AND EVALUATION.

PROCEDURE:

PRE-EXAMINATION, CHARACTERIZATION, AND EVALUATION OF SPECIMEN MATERIAL (1 WEEK), PREPARE SPECIMENS - INSPECT AND ANALYZE SELECTED SPECIMENS AFTER PREPARATION (2 DAYS). SET UP MASS SPECTROMETER AND GAS SAMPLING SYSTEM, HEATING AND FIXTURING FOR LEVITATION EXPERIMENT (1 HOUR). SPECIMEN INSERTION INTO LEVITATION COIL (10 MINUTES). PUMP DOWN TO DESIRED VACUUM LEVEL (20 MINUTES). APPLY POWER TO COIL AND LEVITATE THE SPECIMEN, ADJUST ELECTRONICS TO STABILIZE SAMPLE (20 MINUTES). HEAT SPECIMEN, CONTROL TEMPERATURE (30 MINUTES). FOR SELECTED RUNS, APPLY ELECTRON BEAM HEATING AND ADJUST POWER (10 MINUTES). MONITOR TEMPERATURE AND PARTIAL PRESSURES OF RESIDUAL GASES IN CHAMBER (DURING RUN). HOLD AT SELECTED DWELL TIMES WITH RF HEATING OR RF AND ELECTRON BEAM HEATING AT SELECTED DWELL TEMPERATURES (2 HOURS). IDENTIFY GASEOUS SPECIES WITH MASS SPECTROMETER (DURING RUN). IMPINGE H_2 GAS ON SPECIMEN (OPTION - DURING RUN). COOL IN VACUUM TO ROOM TEMPERATURE (6 TO 20 MINUTES). MATERIALS CHARACTERIZATION AND EVALUATION OF PROCESSED SPECIMENS (1 WEEK).

Figure III-62. Summary Definition of Requirements for Verification Experiments or Tests, 4 - Vacuum Degassing with Levitation

FABRICATION OF TUNGSTEN X-RAY TARGETS

PURPOSE:

TO VERIFY, FOR THE TOTAL PROCESS CYCLE, THE FEASIBILITY OF (A) RF COIL POSITIONING AND HEATING, AND (B) ELECTRON BEAM HEATING AS AN AUXILIARY METHOD OR TO INCREASE THE HEATING EFFICIENCY AND GIVE BETTER TEMPERATURE CONTROL. TO DETERMINE THE EFFECT OF TUNGSTEN EVAPORATION IN A VACUUM AND IN AN INERT GAS. (TO ESTABLISH POSSIBLE NEED FOR INERT GAS DURING HEATING AND MELTING.) TO DETERMINE PROPERTIES, GAS CONTENT, MECHANICAL PROPERTIES, GRAIN SIZE AND SHAPE WITH PROCESSING TIME AS A VARIABLE. TO OBTAIN PRELIMINARY DATA ON SUPERCOOLING OF MOLTEN TUNGSTEN. TO ESTABLISH TIMING OF PROCESS STEPS APPLICABLE TO FURTHER (SHORT DURATION) ZERO "G" EXPERIMENT.

CONDITIONS:

GROUND LABORATORY ENVIRONMENT WITH PROCESSING IN VACUUM OF 10^{-3} TO 10^{-6} N/M^2 AND IN WELL SCRUBBED, HIGH PURITY INERT GAS. TEMPERATURE; FIRST TO DEGASSING TEMPERATURE (1800°C TO 2400°C) AND THEN TO MELTING TEMPERATURE (3410°C). SELECTED DWELL TIME AT DEGASSING TEMPERATURE AND A SHORT DWELL MOLTEN.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

10 GRAMS OR MORE (LIMIT DETERMINED EXPERIMENTALLY) TUNGSTEN SPECIMENS OF 1 CM^3 OR MORE (LIMIT DETERMINED EXPERIMENTALLY).

EQUIPMENT, APPARATUS REQUIRED:

VACUUM SYSTEM $\backsim 10^{-3}$ TO 10^{-6} N/M^2. LEVITATION CHAMBER FOR HIGH VACUUM AND FOR INERT GAS. RF LEVITATION COIL WITH ELECTRONICS FOR POSITION CONTROL AND LATERAL OSCILLATION DAMPING. ELECTRON BEAM UNIT FOR HEATING. RF GENERATOR WITH POWER CAPABILITY FOR 4 KILOWATTS TO 25 KILLOWATTS AT 450 KHZ (4 KW FOR LEVITATION WITH ELECTRON BEAM HEATING, 25 KW FOR LEVITATION WITH RF HEATING). DC POWER SUPPLY FOR ELECTRON BEAM. MASS SPECTROMETER AND GAS SAMPLING SYSTEM. TEMPERATURE MEASURING EQUIPMENT. MATERIALS EVALUATION AND CHARACTERIZATION APPARATUS. VACUUM FUSION GAS ANALYSIS EQUIPMENT. EMISSION SPECTROSCOPE OR SPARK GAP MASS SPECTROMETER. COMPUTER FOR DATA ANALYSIS AND EVALUATION.

PROCEDURE:

PRE-EXAMINATION, CHARACTERIZATION, AND EVALUATION OF TUNGSTEN MATERIAL (1 WEEK). PREPARE SPECIMENS - EXAMINE AND EVALUATE SAMPLES AFTER PREPARATION (2 DAYS). INSERT SPECIMEN AT LEVITATION COIL (MINUTES). EVACUATE CHAMBER (10 MINUTES). IF INERT GAS IS USED, INTRODUCE INTO CHAMBER. (PRESSURE SUFFICIENT TO PREVENT ARCING IN LEVITATION COIL (10 MINUTES). LEVITATE AND STABILIZE SPECIMEN (5 MINUTES). HEAT BY RF AND/OR ELECTRON BEAM IN VACUUM, OR HEAT BY RF ALONE IN INERT GAS (DURING RUN). MONITOR RESIDUAL GAS PARTIAL PRESSURES AND SPECIES; IDENTIFY WITH MASS SPECTROMETER (DURING RUN). MEASURE TEMPERATURE AND TOTAL PRESSURE (DURING RUN). DWELL AT SELECTED TEMPERATURES FOR SELECTED TIMES; IF DETERMINED NECESSARY, INTRODUCE HYDROGEN (2½ HOURS). MELT (5 MINUTES). DWELL 5 TO 10 MINUTES WHEN JUST MOLTEN AND/OR SUPERHEATED AS REQUIRED. COOL BY ELIMINATING ELECTRON BEAM HEATING AND/OR ADJUSTING RF POWER OR FREQUENCY. TRY TO SUPERCOOL (20 MINUTES). AFTER SAMPLE HAS SOLIDIFIED AND BEGUN TO COOL, ELIMINATE RF LEVITATION, DROP ONTO RETRIEVAL CONTAINER (1½ HOUR). MATERIALS CHARACTERIZATION AND EVALUATION (1 WEEK). OPERATIONAL TESTING (15 DAYS).

Figure III-63. Summary Definition of Requirements for Verification Experiments or Tests, 5 - Levitation, Heating, and Melting at One "G"

PURPOSE:

TO VERIFY FEASIBILITY OF POSITIONING DEVICE IN ZERO "G".

CONDITIONS:

ZERO "G" (10^{-3}G) FOR SHORT DURATIONS (3 SECONDS TO 10 MINUTES).
VACUUM (10^{-3}N/M^2) OR INERT GAS (10^{5}N/M^2) AT SPECIMEN.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

SELECTED TUNGSTEN SPECIMENS OF 10 TO 200 GRAMS, ~1 CM^3 TO 10 CM^3.
RF POSITIONING SYSTEM, INCLUDING ELECTRONIC POSITION MONITORING.

EQUIPMENT, APPARATUS REQUIRED:

ALTERNATIVELY, DROP TOWER, K.C.-135, AND/OR SOUNDING ROCKET WITH ADEQUATE POWER SUPPLY AND RF GENERATOR. INSTRUMENTATION, PHOTOGRAPHY TO MEASURE AND RECORD POSITIONING ACCURACY AND STABILITY.

PROCEDURE:

PREPARE SPECIMEN (2 DAYS). INTEGRATE EQUIPMENT INTO TEST PACKAGE FOR DROP TOWER, KC-135, OR SOUNDING ROCKET (1 TO 2 WEEKS). INSTALL TEST PACKAGE INTO DROP TOWER CONTAINER, KC-135, OR SOUNDING ROCKET PAYLOAD HOUSING (1 WEEK). PRE-TEST CHECKOUT AND ESTABLISH TEST CONDITIONS (DROP CHAMBER PUMP-DOWN, KC-135 TAKE-OFF AND ASCENT, SOUNDING ROCKET LAUNCH AND ASCENT (1/2 TO 1 HOUR). ZERO "G" TEST OPERATION (DROP TOWER 3 TO 10 SECONDS, KC-135 20 TO 40 SECONDS, SOUNDING ROCKET 4 TO 10 MINUTES). APPLY POWER TO POSITION AND STABILIZE SPECIMEN, MONITOR SPECIMEN POSITION, HOLD POSITIONG FOR AS LONG AS POSSIBLE, I.E., 3 SECONDS TO 10 MINUTES (DURING RUN). CAGE SAMPLE FOR RECOVERY (~1 SECOND). RE-ENTRY AND DESCENT (SOUNDING ROCKET) (1/2 TO 1 HOUR). RE-SET AND RETEST (KC-135 AND DROP TOWER. RECOVER TEST PACKAGE AND RECORDED DATA.

Figure III-64. Summary Definition of Requirements for Verification or Tests, 6 - Limited Time Zero G Testing of Positioning Devices

PURPOSE:

TO OBTAIN PRELIMINARY DATA ON SPECIMEN POSITIONING IN THE RF COIL, TIME/TEMPERATURE PARAMETERS DETERMINATIONS FOR DEGASSING AND RESULTING TUNGSTEN STRUCTURE AND PROPERTIES.

CONDITIONS:

AS IN 6. FOR SOUNDING ROCKETS.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

SELECTED TUNGSTEN SPECIMEN OF 4 TO 10 GRAMS, SPHEROID OR SLUG. RF POSITIONING AND HEATING DEVICE, ELECTRON BEAM SUPPLEMENTARY HEATING SYSTEM.

EQUIPMENT, APPARATUS REQUIRED:

EXPERIMENT PAYLOAD CONTAINER WITH FIXTURING TO CAGE SAMPLE FOR ASCENT AND DESCENT, GAS MONITOR, INSTRUMENTATION, PHOTOGRAPHY TO MEASURE AND RECORD POSITIONING ACCURACY AND STABILITY, TEMPERATURE OF SPECIMEN, POWER, ETC. POWER SUPPLY. INERT GAS SYSTEM, HYDROGEN SYSTEM. TELEMETRY.

PROCEDURE:

PREPARE SPECIMENS - INSPECT AND TEST SAMPLE SPECIMENS (2 DAYS). INTEGRATE EQUIPMENT INTO TEST PAYLOAD FOR AEROBEE (1 TO 2 WEEKS). INSTALL TEST PAYLOAD INTO AEROBEE PAYLOAD HOUSING (1 WEEK). PRE-TEST CHECKOUT, LAUNCH, ASCENT (1/2 TO 1 HOUR). TEST OPERATIONS (UNCAGE SPECIMEN, APPLY POWER TO POSITION SPECIMEN, INCREASE POWER RATE TO OBTAIN HEATING, ACTIVATE ELECTRON BEAM, MONITOR GAS EFFLUX AND POSITION DURING HEATING, MAINTAIN 1800 C TO 2400 C TEMPERATURE FOR 5 MINUTES, ELIMINATE HEATING BUT MAINTAIN SAMPLE POSITION DURING COOLING. STORE SAMPLE IN INERT ATMOSPHERE FOR DESCENT (1/2 TO 1 MINUTE). RE-ENTRY AND DESCENT (1/2 TO 1 HOUR). RECOVER TEST PACKAGE. GROUND ANALYSIS; COMPOSITION, MECHANICAL PROPERTIES, STRUCTURE (1 WEEK).

Figure III-65. Summary Definition of Requirements for Verification Test, 7 - Limited Time - Zero "G" Heating and Vacuum Degassing

PURPOSE:

TO OBTAIN LIMITED TIME FEASIBILITY VERIFICATION OF THE OVERALL OPERATION OF PROCESS AT ZERO "G".

CONDITIONS:

AS IN 6 FOR SOUNDING ROCKET.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

SELECTED TUNGSTEN SPECIMEN ~10 TO 100 GRAMS, ~1 CM X 1 CM. EQUIPMENT AS IN 7.

EQUIPMENT, APPARATUS REQUIRED:

AS IN 7 WITH INCREASED POWER SUPPLY FOR MELTING.

PROCEDURE:

PROCEDURES PRIOR TO TEST OPERATIONS AS IN 7. TEST OPERATIONS (UNCAGE SPECIMEN, APPLY POWER TO POSITION SPECIMEN WITH RF COIL, INCREASE RF POWER TO HEAT SPECIMEN, ACTIVATE ELECTRON BEAM, DWELL AT DEGASSING TEMPERATURE, ~1800°C TO 2400°C, APPLY HYDROGEN IF NEEDED, INCREASE TEMPERATURE TO NEAR MELT ~3300 C, REMOVE HYDROGEN GAS - ADD INERT GAS IF NEEDED, MELT AND SUPERHEAT > 3400 C, END HEATING AND ATTEMPT TO SUPERCOOL, MAINTAIN SAMPLE POSITION UNTIL COOL ~200°C TO 300°C, CAGE AND STORE SAMPLE IN INERT ATMOSPHERE FOR DESCENT (8 TO 10 MINUTES). RE-ENTRY, DESCENT, RECOVERY, AS IN 7. GROUND TESTS; MECHANICAL PROPERTIES, STRUCTURE, GAS CONTENT, DENSITY, DBTT, ETC. (1 WEEK).

Figure III-66. Summary Definition of Requirements for Verification Test, 8 - Limited Time Zero "G" - Degassing, Melting and Supercooling Process

PURPOSE:

TO ESTABLISH ALL PROCESS DESIGN PARAMETERS FOR EQUIPMENT TO PRODUCE THE HIGH PURITY TUNGSTEN PRODUCT. TO ENSURE OPERATIONAL PERFORMANCE AT ZERO "G" FOR EXTENDED TIME.

CONDITIONS:

LONG DURATION (HOURS) OF ZERO "G", CONDITIONS AT SPECIMENS AS IN 8.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

10 TO 200 GRAM TUNGSTEN SPECIMENS OF VARIOUS PURITIES, 1 CM^3 TO 10 CM^3, DEVELOPMENT VERSIONS OF PROCESSING CHAMBER, POSITIONING AND RF HEATING SYSTEM, ELECTRON BEAM HEATING SYSTEM, INERT GAS AND HYDROGEN GAS SYSTEMS PROCESS INSTRUMENTATION. TEMPERATURE, GAS PRESSURE INSTRUMENTATION. TEST CHECKOUT SUPPORT.

EQUIPMENT, APPARATUS REQUIRED:

SHUTTLE SORTIE LABORATORY, ELECTRICAL POWER (4 TO 7 KW), TEST MONITORING STATION.

PROCEDURE:

PREPARE SPECIMENS - SELECTED QUALITIES (1 WEEK). INTEGRATION OF EQUIPMENT INTO SORTIE LAB AND TRAINING OF TEST OPERATOR (3 TO 6 MONTHS). PRE-LAUNCH PROCEDURES (1 TO 2 WEEKS). ASCENT AND ESTABLISHMENT OF ORBIT (1½ HOURS). CHECKOUT EXPERIMENT EQUIPMENT, TEST OPERATIONS; INSERT SPECIMEN, APPLY POWER TO POSITION SPECIMEN WITH RF COIL (5 TO 10 MINUTES). STABILIZE SPECIMEN, INCREASE RF POWER FOR HEATING, ACTIVATE ELECTRON BEAM FOR ADDITIONAL HEAT AS REQUIRED (DURING RUN), DWELL TIME AT SELECTED TEMPERATURES FOR DEGASSING (1 TO 2 HOURS). APPLY PARTIAL PRESSURE OF INERT GAS IF NEEDED, APPLY HYDROGEN IF REQUIRED, INCREASE TEMPERATURE FOR ADDITIONAL DEGASSING IF NECESSARY (DURING RUN), MELT USING PARTIAL PRESSURE OF GAS IF NEEDED TO CONTROL EVAPORATION (1 MINUTE), SUPER-HEAT (5 TO 10 MINUTES). SUPERCOOL (10 MINUTES TO 1 HOUR). COOL (5 TO 20 MINUTES). STORE SAMPLE (2 MINUTES). DESCENT, RE-ENTRY, AND RECOVERY (IN SHUTTLE SEQUENCE). GROUND TESTS; MECHANICAL PROPERTIES, STRUCTURE, GAS CONTENT, DBTT, DENSITY, ETC. (1 WEEK PER SPECIMEN). OPERATIONAL TESTING (15 DAYS).

Figure III-67. Summary Definition of Requirements for Verification Test, 9 - Establishment of Final Process Design Parameters

FABRICATION OF TUNGSTEN X-RAY TARGETS

PURPOSE:

TO PROOF-TEST THE FINAL TUNGSTEN X-RAY TARGET PROCESSING SYSTEM. TO OBTAIN DEFINITION OF PRODUCTION PARAMETERS, PROCESSING RATE, QUALITY, ETC.

CONDITIONS:

AS IN 9.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

SUFFICIENT BILLETS OF TUNGSTEN FOR 1 TO 5 DAYS PRODUCTION, AND OF QUALITY AND SIZE SELECTED FROM PREVIOUS TESTING; FLIGHT QUALIFIED PROCESSING SYSTEM (CHAMBER, INSERTION AND RETRIEVAL DEVICE, RF COIL AND ASSOCIATED ELECTRONICS, ELECTRON BEAM GUN AND ASSOCIATED ELECTRONICS, INERT GAS SYSTEM AND HYDROGEN SYSTEM IF INDICATED FROM PREVIOUS TESTING, PROCESS INSTRUMENTATION, PROCESS CONTROL CENTER).

EQUIPMENT, APPARATUS REQUIRED:

SHUTTLE SORTIE LAB, ELECTRICAL POWER SUFFICIENT FOR PROOF-TEST RUN, THERMAL CONTROL, COMMUNICATIONS, TEST INSTRUMENTATION DATA MANAGEMENT.

PROCEDURE:

PRE-TEST AS IN 9. INITIATE AUTOMATED PROCESSING, PERFORM MANUAL FUNCTIONS AS REQUIRED. MONITOR PROCESS AND TEST INSTRUMENTATION. PERFORM MAINTENANCE AND REPAIR. SHUT DOWN AND STOW PRODUCTS ON COMPLETION OF TEST (5 TO 7 DAYS). POST-TEST AS IN 9. OPERATIONAL TESTING OF PRODUCTS (15 DAYS).

Figure III-68. Summary Definition of Requirements for Verification Test, 10 - Prototype Demonstration

FABRICATION OF TUNGSTEN X-RAY TARGETS

DESCRIPTION:

TUNGSTEN SAMPLES OF SUFFICIENT SIZE PREPARED BY A PRECEDING EXPERIMENT OR TEST WILL FIRST BE METALLURGICALLY CHARACTERIZED AND EVALUATED. WHERE ENHANCEMENT IN MECHANICAL PROPERTIES WARRANTS, SAMPLES WILL BE TESTED OPERATIONALLY IN X-RAY TUBES BUILT FOR THAT PURPOSE. THESE TESTS WILL ESTABLISH WHETHER THE MATERIAL PRODUCED CAN BE FABRICATED INTO A TARGET WHICH HAS INCREASED EXPOSURE LIFE, THE AMOUNT OF X-RADIATION FALL-OFF AFTER THE EXPOSURES, AND WARPAGE RESISTANCE OF THE TARGET.

PURPOSE:

TO DETERMINE WHETHER THE PROCESS CAN PRODUCE TUNGSTEN X-RAY TARGETS WITH THE DESIRED ENHANCEMENT OF OPERATIONAL CAPABILITIES. TO HELP PREDICT THE IMPROVEMENT IN SERVICE PROPERTIES WHICH MAY BE OBTAINED BY PROCESSING IN THE WEIGHTLESS ENVIRONMENT OF SPACE.

CONDITIONS:

GROUND LABORATORY EQUIPMENT WITH X-RAY TUBE OPERATIONAL CONDITIONS.

SPECIMENS, SAMPLES TO BE TESTED:

DEPENDING ON QUANTITY OF TUNGSTEN PRODUCED IN PRECEDING TEST, SPECIMEN BONDED ONTO CONVENTIONAL TARGET OR FABRICATED INTO COMPLETE TARGET.

APPARATUS:

CONVENTIONAL X-RAY TESTING CHAMBER WITH TESTING TUBE, INSTRUMENTATION FOR FOCAL SPOT DIAMETER, X-RAY INTENSITY, X-RAY PATTERN, ETC., AUTOMATED LIFE TEST SWITCHING AND POWER SUPPLY, LIFE TEST SOFTWARE. TARGET EVALUATION AND CHARACTERIZATION EQUIPMENT.

PROCEDURE:

FABRICATE A BUTTON OR TARGET FROM THE PRESSED MATERIAL AND, WHERE NECESSARY, BOND IT TO A STANDARD X-RAY TARGET (3 DAYS). INCORPORATE THE TARGET INTO AN X-RAY TUBE (2 DAYS). SET UP X-RAY MACHINE AND TEST EQUIPMENT (1/2 DAY). PERFORM LONG LIFE EXPOSURE TEST (30,000 EXPOSURES AT 150 KVP AND 250 MA WITH 1.25 SECOND EXPOSURES TAKEN AT THE RATE OF TWO EXPOSURES PER MINUTE ON A 2 MM FOCAL SPOT (11 DAYS). REMOVE TARGET, ANALYZE SAMPLE.

Figure III-69. Operational Testing for Evaluation of Tungsten Material as X-Ray Target Material

III.7.3 SUMMARY OF KNOWLEDGE GAPS, EXPERIMENT AND TEST REQUIREMENTS FOR FABRICATING HIGH PURITY TUNGSTEN X-RAY TARGETS

A summary version of knowledge gaps discussed earlier, together with the related Experiments and Tests, and with their requirements is given in Figures III-70A and III-70B.

KNOWLEDGE GAPS	EXPERIMENTS AND VERIFICATION TESTS	EXPERIMENT AND TEST REQUIREMENTS (SUMMARY)
DEGASSING RATES OF VARIOUS IMPURITIES. EFFECTS OF TEMPERATURE, VACUUM AND GAS PARTIAL PRESSURES, INITIAL TUNGSTEN PURITY	INITIAL VACUUM DEGASSING EXPERIMENTS ON; DEGASSING TIMES VS TEMPERATURE, TYPE AND QUANTITY OF GASES AND OTHER IMPURITIES, DEGASSING EFFECTS ON MECHANICAL PROPERTIES, INTERSTITIALS CONTENT, GRAIN SIZE AND SHAPE, TO AID IN SELECTION OF INITIAL TUNGSTEN QUALITY.	STANDARD GROUND LAB WITH VACUUM INDUCTION FURNACE, RF GENERATOR, MASS SPECTROMETER, GAS SAMPLING SYSTEM, PROCESS INSTRUMENTATION, MATERIALS AND GAS ANALYSIS EQUIPMENT, SPECTROSCOPE. SPECIMENS OF GRADES OF TUNGSTEN. 2-1 2 WEEKS PER SPECIMEN. MANUAL CONTROL.
DESIGN DATA FOR POSITIONING, CONTROL RF COIL	INITIAL SOLID TUNGSTEN POSITIONING, CONTROL EXPERIMENTS TO DETERMINE RF COIL DESIGN, POWER REQUIREMENTS.	STANDARD GROUND LAB, RF GENERATOR, POSITIONING CONTROL COIL, VACUUM CHAMBER, OPTICAL PYROMETER, TUNGSTEN SPECIMENS - 10G TO 100G. PHOTOGRAPHIC EQUIPMENT. PROCESS INSTRUMENTATION. 3-1 2 HOURS PER RUN. MANUAL.
SELECTION AND DESIGN DATA FOR TUNGSTEN HEATING SYSTEM. EFFECTS OF INITIAL TUNGSTEN PURITY.	EXPERIMENTS ON ELECTRON BEAM HEATING OF TUNGSTEN TO ESTABLISH HEATING EFFICIENCY AND AID IN SELECTION OF INITIAL TUNGSTEN QUALITY.	STANDARD GROUND LAB, ELECTRON BEAM GUN, X-RAY PROTECTION, VACUUM CHAMBER, 10KV 1-2 AMP POWER SUPPLY, SPECIMEN FIXTURING, TEMPERATURE AND POWER INSTRUMENTATION, MASS SPECTROMETER, SPECIMENS OF TUNGSTEN VARIOUS GRADES , MATERIALS ANALYSIS EQUIPMENT. 4 TO 5 HOURS PER SPECIMEN. MANUAL.
SELECTION OF SYSTEM OR COMBINATION OF SYSTEMS FOR HEATING, POSITIONING OF TUNGSTEN. ASSESSMENT OF INTERACTIONS FOR COMBINATION SYSTEM. EFFECTIVENESS OF DEGASSING. EFFECTS OF PRE-MELT DWELL AND INITIAL TUNGSTEN PURITY.	EXPERIMENTS ON VACUUM DEGASSING WITH LEVITATION TO COMPARE RF POSITIONING AND HEATING VS. RF POSITIONING WITH ELECTRON BEAM HEATING FOR DEGASSING STEP. ALSO DETERMINE AMOUNT AND TYPE OF CONTAMINANT RELEASE AS FUNCTION OF HEATER AND DWELL TIMES. AID IN SELECTION OF INITIAL TUNGSTEN PURITY.	STANDARD GROUND LAB WITH VACUUM SYSTEM AND LEVITATION CHAMBER, RF LEVITATION POSITIONING COIL, ELECTRON BEAM GUN, VARIOUS PURITY TUNGSTEN SPECIMENS, SPECIMEN FIXTURING, RF POWER SUPPLY (4 KW AT KHZ TO 25 KW AT 450 KHZ), MASS SPECTROMETER, GAS SAMPLING SYSTEM, PROCESS INSTRUMENTATION, METALLURGICAL CHARACTERIZATION. 4-1/2 HOURS PER RUN. MANUAL.
ASSESSMENT OF POSITIONING, HEATING, DEGASSING, MELTING, SUPERCOOLING ADVANTAGES. PRELIMINARY SELECTION OF PROCESS ATMOSPHERE. PRELIMINARY ASSESSMENT OF PROCESSED TUNGSTEN METALLURGICAL AND OPERATIONAL PROPERTY GAINS.	LEVITATION, HEATING, MELTING TEST FOR FEASIBILITY OF R F AND OR ELECTRON BEAM AS CONTROL AND HEATER. ALSO ASSESS EFFECTS OF VACUUM AND INERT GAS ON TUNGSTEN EVAPORATION. ALSO PRELIMINARY SUPERCOOLING EXPERIMENT.	STANDARD GROUND LAB. EQUIPMENT AS ABOVE PLUS INERT GAS SYSTEM AND COPPER OR CERAMIC COOLING CRUCIBLE. ALSO OPERATIONAL TESTING (TEST X-RAY TUBES, PROGRAMMED TEST APPARATUS, STANDARD SHIELDED TEST CHAMBER, ETC). 4-1/2 TO 5 HOURS PER RUN PLUS 1 WEEK CHARACTERIZATION PLUS 15 DAYS OPERATIONAL TESTING. MANUAL PLUS AUTOMATED OPERATIONAL TESTING.

Figure III-70A. Definition of Requirements for Experiments to Verify Selected Approach for Processing High Purity Tungsten X-Ray Targets

KNOWLEDGE GAPS	EXPERIMENTS AND VERIFICATION TESTS	EXPERIMENT AND TEST REQUIREMENTS (SUMMARY)
ASSESSMENT OF POSITIONING SYSTEM AND ACQUISITION OF DESIGN DATA.	ZERO "G" VERIFICATION TEST OF POSITIONING SYSTEM.	DROP TOWER, KC135, OR SOUNDING ROCKET. TEST PACKAGE OF POSITIONING DEVICE, TUNGSTEN SPECIMEN, CAGING SYSTEM, POSITION MONITORING INSTRUMENTATION, RECORDER, POWER SUPPLY AND DISTRIBUTION, PHOTOGRAPHIC APPARATUS, MEASUREMENT GRID, AUTOMATED. MINIMUM TEST TIME 4 TO 10 SEC – DROP TOWER, 20 TO 40 SEC – KC135, 4 TO 10 MIN.- SOUNDROCKET. TELEMETRY OR RECORDING OF DATA, RECOVERY OF TEST PACKAGE AND RECORDER DATA.
EFFECTIVENESS OF HEATING AND DEGASSING METHODS	ZERO "G" PRELIMINARY VERIFICATION TESTS OF HEATING AND DEGASSING TECHNIQUE.	SOUNDING ROCKET. TEST PACKAGE AS ABOVE PLUS ELECTRON BEAM GUN, INCREASED POWER SUPPLY, GAS SAMPLING SYSTEM AND ANALYZER, INERT GAS SYSTEM. AUTOMATED. MINIMUM TEST TIME, 7 TO 10 MINUTES. TELEMETRY OR RECORDING OF DATA, RECOVERY OF TEST PACKAGE, PROCESSED SAMPLE, RECORDED DATA.
ASSESSMENT OF PROCESS EFFECTIVENESS	ZERO "G" PRELIMINARY VERIFICATION TESTS OF DEGASSING, MELTING, SUPERCOOLING TECHNIQUE	SOUNDING ROCKET. TEST PACKAGE AS ABOVE PLUS FURTHER INCREASED POWER SUPPLY, MAXIMUM SIZE SPECIMEN. AUTOMATED, MINIMUM TEST TIME 8–10 MINUTES. TELEMETRY AND RECOVERY AS ABOVE.
FINAL PROCESS DESIGN DATA DEFINITIONS	ZERO "G" PROCESS VERIFICATION TEST	SHUTTLE FACILITY. TEST PACKAGE AS ABOVE, AUGMENTED FOR MULTIPLE SAMPLES, EXTENDED TEST TIMES, AND COMPLETE PROCESS INSTRUMENTATION. PROVISION FOR CREW OBSERVATION, MONITORING AND PROCESS MODIFICATION. AUTOMATED PROCESS, MANUAL RECYCLE. 1 1/2 – 3 HOURS PER RUN, PLUS 15 DAYS OPERATIONAL TESTING. TELEMETRY OF DATA, RECOVERY OF TEST PACKAGE AND PROCESSED SAMPLES.
PRODUCTION LINE DEFINITION	PROTOTYPE FACILITY DEMONSTRATION	SHUTTLE FACILITY. PROCESSING LINE INCORPORATING SELECTED POSITIONING, HEATING, COOLING, ATMOSPHERE AND INITIAL TUNGSTEN QUALITY RESULTING FROM PRECEDING TESTS AND EXPERIMENTS. POWER AND AUTOMATION FOR PROVIDING TYPICAL PRODUCTION QUANTITY, QUALITY, TIMING. AUTOMATED. 5 TO 7 DAYS PLUS 15 DAYS OPERATIONAL SYSTEMS. RECOVERY OF ALL PRODUCT SAMPLES, PRODUCTION EQUIPMENT.

Figure III-70B. Definition of Requirements for Experiments to Verify Selected Approach for Processing High Purity Tungsten X-Ray Targets

III.8 DEFINITION OF REQUIREMENTS FOR EXPERIMENTS TO VERIFY SELECTED APPROACH FOR FABRICATION OF SURFACE ACOUSTIC WAVE COMPONENTS

III.8.1 CRITICAL ELEMENTS IN SELECTED APPROACH FOR FABRICATION OF SURFACE ACOUSTIC WAVE COMPONENTS, AND RELATED KNOWLEDGE GAPS

As pointed out earlier (Section III.4.1), the two major problems, which encompass the critical elements of the selected approach, are (1) obtaining crystals with the required degree of perfection, and (2) imprinting the extremely fine circuitry on the substrate crystal wafers.

Reviewing the steps in the selected approach Figure III-30, enables the identification of the key process elements which affect these problems, and for which additional information is needed.

III.8.1.1 GROWTH OF CRYSTALS (FOR SUBSTRATES)

Lithium Niobate and Sapphire have been selected from examples of piezoelectric and non-piezoelectric materials which exhibit a degree of advantages over other substrate materials. Nevertheless, final selection will depend on specific applications and relative success in producing high quality crystals of suitable size.

Converting these materials into crystals of sufficient purity and perfection for use in Surface Acoustic Wave component substrates is a step in the process under study which can tolerate little or no compromises. The previously discussed crystal quality requirements will stretch the capabilities of the crystal-growing art to its utmost. Fortunately, crystal growth has been identified in earlier studies as an area in which space processing might offer significant advantages, and work has already begun on various aspects of growth techniques, environmental effects, etc. While such efforts have yet to prove the advantages of crystal

rowth in space, a number of analyses and experiments have provided initial nformation, or at least techniques, for characterizing the effects of space nvironment on crystal growth, Typically, such studies include the Skylab experment M512 on Gallium Arsenide, National Bureau of Standards analytical and xperimental programs on convection effects and crystal growth, and various ndustry programs. Typical related reports are listed below; (1), (2), (3).

t is important to note that, while the process for fabrication of Surface coustic Wave Components requires high perfection crystals, it is not a requirement that these crystals be grown in space. Further development of this process ust provide for close surveillance of crystal growing experiments, whether they e ground- or orbit-based, whether they be Czochralski, flux-grown, or some daptation of these, and should include vapor-phase Epitaxial growth (for use ith non-piezo substrates).

Ulrich (Appendix C, Volume II, Book 2) points out a number of types of imperfections hat occur in ground-grown crystals. The Czochralski technique applied to Lithium iobate, tends to induce compositional differences along the crystal length and rom crystal to crystal. Spiral radial strain patterns have also been observed. lux-growth tends to produce Lithium Niobate crystals with micro-cracks. It emains to be shown, therefore, whether modified versions of these techniques

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(1) Ulrich, D. R.; Noone, M. J.; White, W. B; and Henry, E. G.: Crystal Growth in Fuxed Solvent Systems; General Electric Company; Final Report, Contract NAS8-28114, June, 1973.

(2) Padovani, F. A.; Voltmer, F. W.: Growth of a Single Crystal Ribbon in Space; Texas Instruments Incorporated; Contract NAS8-27807, 1973.

(3) Parket, H. S.; Dragoo, A. L.: Investigation of Convective Effects and Crystal Growth; National Bureau of Standards; Contract #W-13475, 1973.

operated in the zero "G" of orbital flight can alleviate these problems by eliminating or minimizing convective flows.

The potential advantages of specific non-piezoelectric materials as crystal substrates for some applications warrants retaining the optional process step of depositing a piezo crystal film (without which such substrates could not function as Surface Acoustic Wave devices) on the substrates. It is conjectured that epitaxial deposition of such films in zero "G" via a diffusion process will minimize non-uniformities in the crystal film arising from convective flows and loss of saturation in the vapor adjacent to the substrate. The extent of such benefits has not been proved, and data are needed to determine whether the benefits might not be nullified by loss of the mixing due to convection.

Final decisions on crystal growing await acquisition of data in the following areas:

- o Identification of the most beneficial crystal materials (based on applications, cost).
- o Determination of the best crystal growth method (based on largest size, quality, surface/plane alignment).
- o Assessment of the effects of zero "G" on epitaxial crystal film growth.

III.8.1.2 Fabrication of Masks

This is the most critical process step in the selected fabrication approach. Without lithographic masks of the most exacting accuracy (of the order of $\pm$20-25A^{o}) viable production of Surface Acoustic Wave Components capable of operation in the 10 to 30 GHz range cannot be accomplished. Earlier discussion (Section III.4.1.3) has cited the importance and effects of low frequency vibrations in this process, thus establishing that knowledge of the vibration environment in which mask fabri-

cation is to take place is mandatory. Appendix D in Volume II, Book 2, discusses this program in further detail, noting the vibration levels in ground facilities, and the attempts to reduce such levels with a seismic isolator pad. Appendix D also provides a sample of the in-orbit vibration data available, and indicates that the less than 20 micro-G at a frequency of 7.5 Hz reflects the general possibility that in-orbit conditions will enable the desired electron beam mask fabrication. This appendix also notes some concepts for carrying out this process step. More definitive data on in-orbit vibrations (more accurate G levels, frequencies of $\sim$.1 to .01 Hz), and on isolation methods are needed.

In addition to the vibration problem, a key gap in the existing knowledge is whether electron beams can "cut" lithographic masks with line widths of 200-250A°, and how accurate the lines will be. The energy distribution within the 100A° wide beam will make it difficult to attain the required accuracy. Furthermore, the combined effects of vibration and electron beam accuracy on mask fidelity must be assessed, as must the effects of the $\sim$40 hour period required to fabricate a mask.

In summary, definitive data on Mask Fabrication is lacking on the following points:

- o Vibration levels at various positions on various spacecraft. These data will form the basis for a comprehensive method of predicting effects on, and isolation methods for, Mask Fabrication.
- o Electron Beam Gun accuracy in Mask Fabrication, and effects of vibrations and vibration isolation on such fabrication.

III.8.1.3 Lithographic Exposure

In this process step, the mask is used as a stencil and soft x-rays (from an electron beam focussed on an aluminum target) sweep the mask, reacting with the resist coating on the crystal substrate wherever the mask has openings. In general, such an exposure requires a duration of about 1/2 hour, during which,

in order to attain the high accuracy of the final circuitry, all disturbances should be minimized. The level of tolerable disturbance is not known at this time, and, as noted earlier, data on the vibration levels in spacecraft is needed.

Furthermore, since the process under study is aimed at commercial use, key information is needed on the relationships among costs to achieve tolerable vibration levels, payload weights for x-ray lithographic equipment, and quality (accuracy, repeatability) of final S.A.W. circuitry.

Although soft x-ray lithography was selected as the process step for forming the S.A.W. circuitry on the resist coating of the crystal substrate, direct electron beam "writing" of circuitry on an electron resist coating of the substrate should not be overlooked. In this approach, the electron beam is utilized directly on the electron-resist-coated substrate in a programmed pattern where it reacts with the electron resist to trace the S.A.W. circuitry. There is no intermediate "cutting" of masks as in the previously discussed x-ray lithographic process. The programmed electron beam approach may not be economically feasible for high volume production, due to its lengthy duration (each typical S.A.W. device would require approximately 40 hours, as opposed to the 40 hours per mask and 1/2 hour per device in the x-ray lithographic process. Yet, if data proves the accuracy and fineness of circuitry obtained by the programmed electron beam process, it may be economically feasible for low volume, specialized, high value applications.

Thus, this key element of fabricating Surface Acoustic Wave Components is in need of data on:

- Effects of orbital conditions, vibration levels, isolation methods on x-ray lithographic exposure quality, accuracy, timing. Effects of technique variations on costs, equipment.

- o Performance of programmed electron beam as circuitry "writer" under orbital conditions, especially vibration levels and various isolation methods. Effects of "writing" duration. Cost, quality, equipment data.

I.8.1.4 Other Possible Process Steps

noted in Section III.4.3, within the selected approach for fabrication of rface Acoustic Wave Components, a number of process steps are annotated as referred Performed in Space" (PIS) in Figure III-3D. This notation identifies ocess steps which could be performed on the ground, but which the User would efer to perform in space if economically feasible. These preferences generally flect either the possibility that some secondary technical advantages can crue from the vacuum of space or that handling is minimized by performing ntiguous process steps in space. The following areas fall into this category, d their associated knowledge gaps should be addressed:

bstrate Ultra-Cleaning - Both ion beam scrubbing and back sputtering have been ed in ground processing, and cost of space processing will be a key criterion the final decisions for this process step, if quality of results are equivalent. formation is required as to techniques, equipment requirements and environment fects on the cleanliness achieved by these alternatives.

bstrate Surface Metallization - Again, two presently used methods are possible; uttering and vapor deposition. As above, cost is a key decision factor, while plicability of ground-based technology and quality of results under space con- tions remain the chief unknowns.

plication of Resist Coating - Application through use of the ground-based "spinner" chnique appears to remain applicable, although some modification in technique may required to accommodate zero "G". Cost and quality of results remain key cision factors on this process step, also.

III.8.2 EXPERIMENT REQUIREMENTS FOR VERIFICATION OF SELECTED APPROACH FOR FABRICATION OF SURFACE ACOUSTIC WAVE COMPONENTS

The preceding discussion has acknowledged that considerable additional information is required in order to implement the selected process. Much of that information must be obtained by experiments and tests, while the rest will result from analytical and design studies which require data from experiments.

While the two major problems addressed by experiments and test discussed herein are the acquisition of sufficiently high quality crystals and the formation of the fine, accurate electrode circuitry, the following experiments and tests also reflect the User's desire to evaluate the feasibility of carrying out other process steps in orbit. The data provided by the latter experiments and test will enable the User to make the necessary assessments of both technical and economic feasibility.

The experiment series dealing with crystal growth has been established to provide data in two areas:

- o Evaluation of Ground-Grown Crystals
- o Development Space-Growth Techniques (including the possibility of growth with a principal plane on an exposed surface).

Since considerable effort is already being expended in these areas, the defined experiment series given herein provides only an overview of what the User requires

In the process steps involved in imprinting electrode circuitry, the nominal technique calls for one critical step which appears to require space processing - fabrication of the lithography mask, and a second which testing may pinpoint as critical - the x-ray exposure of resist-coated substrates with the mask.

Another mode of imprinting circuitry, however, has also been discussed earlier - the direct writing of circuitry on the coated substrate with an electron beam. While limited in commercial applicability, the potential technical value of such a mode warranted experimental investigation of its capabilities.

There is a high degree of commonality in required data, test conditions, test equipment, and evaluation procedures for defining the processing steps of imprinting circuitry, and for assessing x-ray lithography versus direct electron beam writing. On that basis, several of the experiments and tests documented herein reflect combined procedures related to two or more process steps.

Figures III-71 through III-78 summarize the requirements for eight series of tests, which it is believed will enable firm definition of a commercial process for fabrication of Surface Acoustic Wave Components which can operate in the 10 to 30 GHz range.

III.8.3 SUMMARY OF KNOWLEDGE GAPS, EXPERIMENT AND TEST REQUIREMENTS FOR FABRICATION OF SURFACE ACOUSTIC WAVE COMPONENTS

A summary of the knowledge gaps discussed earlier, their related experiments and tests, and requirements for such experiments and tests is given in Figures III-79A and III-79B.

PURPOSE:

TO DETERMINE EFFECTS OF ~100 A° ELECTRON BEAM SPOT ENERGY DISTRIBUTION ON MASK EDGE SHARPNESS, AND TO PROVIDE DATA FOR PREDICTING GENERATION OF MASKS WITH SUFFICIENT RESOLUTION. EXPERIMENT DATA SHOULD PROVIDE BASIS FOR PROJECTING 250 A° ELECTRODE FINGER WIDTHS (FOR FREQUENCY OF 30 GHz) AS LIMITED BY MASK RESOLUTION.

CONDITIONS:

STANDARD LABORATORY CONDITIONS WITH ORDINARY MECHANICAL (VIBRATIONAL) STABILITY AND AIR CONDITIONED ENVIRONMENT.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

COMMERCIALLY AVAILABLE CRYSTAL SAMPLES, EITHER QUARTZ OR LITHIUM NIOBATE, WITH EXCELLENT SURFACE, POLISHED AND FREE FROM DEFECTS, 10 CM LONG, BY 2 CM WIDE, BY 2 MM THICK, AND WITH POLYMETHYMETHACRILIATE ELECTRON RESIST SURFACE COAT. 2" X 2" GLASS CARRIERS FOR MASKS.

EQUIPMENT, APPARATUS REQUIRED:

HIGH RESOLUTION (<100 A° SPOT SIZE) ELECTRON BEAM EQUIPMENT FOR HIGH-RESOLUTION WRITING ON ELECTRON RESIST, WITH SIMPLE PRECISION SPOT CONTROLS. BEAM POWER SUFFICIENT FOR RESIST EXPOSURE. SCANNING ELECTRON MICROSCOPE FOR EVALUATION OF EDGE RESOLUTION WILL BE NECESSARY.

PROCEDURE:

PREPARE ELECTRON RESIST LAYERS WITH SUFFICIENT UNIFORMITY AND THIN SECTION FOR 250 A° LINE WRITING. PREPARE SAMPLE HOLDERS FOR WRITING SIMPLE REPETITIVE PARALLEL LINE STRUCTURES, FOR USE WITH THE ELECTRON BEAM WRITING EQUIPMENT. ADJUST THE ELECTRON BEAM EQUIPMENT FOR WRITING WITH A NOMINAL SPOT SIZE AS SMALL AS POSSIBLE, AND NOT LARGER THAN 100 A°. WRITE SIMPLE LINE PATTERNS. EVALUATE WITH SCANNING ELECTRON BEAM. REPEAT, WITH DEVELOPMENT DIRECTED TOWARDS PRODUCING TECHNIQUES WITH BEST RESOLUTION. EVALUATE RESULTANT PATTERN RESOLUTION, AND PARTICULARLY EDGE SHARPNESS. CORRELATE WITH BEAM SHARPNESS AND ENERGY DISTRIBUTION DATA FOR BEAM USED. EVALUATE EFFECTS OF GRADED BEAM EDGE SHARPNESS ON ARRAY PERFORMANCE.

Figure III-71. Summary Definition of Requirements for Verification Experiments or Tests, 1 - Resolution of Electron Beam for Use in SAW Mask Fabrication

FABRICATION OF SURFACE ACOUSTIC WAVE COMPONENTS

PURPOSE:

TO EVALUATE, AND, IF NECESSARY, DEVELOP CRYSTAL GROWING TECHNIQUES AND SELECT MATERIALS WHICH MAXIMIZE FIGURE OF MERIT AND QUALITY (SIZE, PURITY, PERFECTION) OF SUBSTRATES FOR HIGH FREQUENCY S.A.W. DEVICES. (EVALUATION IS FOR CRYSTALS PROVIDED BY TECHNIQUES DEVELOPED IN CURRENT AND PLANNED PROGRAMS. IF CURRENT AND PLANNED TECHNIQUES UNSATISFACTORY OR DO NOT INCLUDE CRYSTALS WITH WORKING SURFACE GROWN ALONG PRINCIPAL PLANE, THIS PROGRAM SHOULD DEVELOP SUCH METHOD).

CONDITIONS:

GROUND LABORATORY; STANDARD CONDITIONS. FOR TECHNIQUE DEVELOPMENT; GROWING TEMPERATURES, PRESSURES, ATMOSPHERES, LACK OF VIBRATION CONSISTENT WITH SPECIFIC MATERIALS AND TECHNIQUES (E.G., $LiNbO_3$ - 1533°K, ONE ATMOSPHERE OF AIR). FOR EVALUATION; ROOM AND CRYOTEMPERATURES AS REQUIRED FOR CHARACTERIZATION METHOD, TEMPERATURES FOR POLING (E.G., 1200°C AT ONE VOLT/CM FOR LITHIUM NIOBATE). IN ORBIT GROWTH; ZERO 'G', MINIMUM VIBRATION. TEMPERATURE, PRESSURE FOR SELECTED MATERIAL AND METHOD.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

FOR GROWTH TECHNIQUE DEVELOPMENT; RAW STOCK/POLYCRYSTALLINE MATERIALS LITHIUM NIOBATE, SAPPHIRE, SPINEL, LITHIUM TANTALATE, BISMUTH GERMANATE FOR SELECTED CRYSTALS. FOR CRYSTAL EVALUATION; CENTIMETER SIZE CRYSTALS AS BASIC SUBSTRATES. AS REQUIRED, WITH THIN FILMS. FOR EVALUATION, RAW UNCUT CRYSTAL SPECIMENS AS GROWN, ALSO CUT, AND CUT AND POLISHED.

EQUIPMENT, APPARATUS REQUIRED:

DEVELOPMENT APPARATUS FOR CRYSTAL GROWING VIA CZOCHRALSKI, FLUX-GROWTH TECHNIQUES, MODIFICATIONS OF THESE, OR NEW TECHNIQUES. ENVIRONMENTAL INSTRUMENTATION AND PHOTOGRAPHY CRYSTAL SAWING, POLISHING, ION BEAM AND BACK SPUTTERING EQUIPMENT, SCANNING ELECTRON MICROSCOPE AND PROBE, X-RAY CRYSTALLOGRAPHY EQUIPMENT, AC AND DC RESISTIVITY AND HALL EFFECT EQUIPMENT, PHOTOLUMINESCENCE EQUIPMENT, SURFACE ANALYZERS.

PROCEDURE:

FOR CRYSTAL-GROWTH TECHNIQUE DEVELOPMENT; GROW CRYSTALS OF CANDIDATE MATERIALS BY ONE OR MORE CANDIDATE TECHNIQUES (1 DAY TO 1 WEEK PER CRYSTAL, DEPENDING ON TECHNIQUE AND SIZE REQUIRED). COLLECT ENVIRONMENT DATA AND PHOTOGRAPHS DURING GROWTH. FOR ORBITAL GROWTH, TELEMETER DATA TO GROUND.

FOR EVALUATION (CRYSTALS OBTAINED FROM ANY CANDIDATE SOURCE) PERFORM EXAMINATION OF EACH CRYSTAL BEFORE AND AFTER EACH OF FOLLOWING STEPS WITH ELECTRON MICROSCOPE, X-RAY CRYSTALLOGRAPHY, AND OTHER CHARACTERIZATION EQUIPMENT TO ASSESS PERFECTION OF CRYSTAL (1 DAY PER CRYSTAL). ORIENT CRYSTAL, WHERE REQUIRED (ONE HOUR). CUT AND POLISH, IF REQUIRED, (TWO TO THREE DAYS). POLE (6 HOURS) IF REQUIRED.

Figure III-72. Summary Definition of Requirements for Verification Experiments or Tests, 2 - Crystal Substrate Growth Method Evaluation & Development

PURPOSE:

TO DEVELOP METHODS AND QUALITY OF FINAL CLEANING OF SUBSTRATE SURFACES IN SPACE ENVIRONMENT.

CONDITIONS:

IN GROUND LABORATORY AND KC135; STANDARD CONDITIONS, SEALED ION BEAMS AND SPUTTERING CHAMBER (10^{-3} TO 10^{-6} N/M^2). IN SOUNDING ROCKET; AMBIENT VACUUM. IN KC135 AND SOUNDING ROCKET; ZERO "G" TRAJECTORY.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

CRYSTALS OF LITHIUM NIOBATE, SAPPHIRE, QUARTZ; FROM 1 MM TO 20 CM IN SIZE (TOTAL 1 KG). ONE SURFACE OPTICALLY FLAT; HANDLING METHODS MUST INSURE FREEDOM FROM SCRATCHING.

EQUIPMENT, APPARATUS REQUIRED:

PRE-CLEANING CHEMICAL CLEANING BATHS, ULTRASONIC CLEANER. ULTRA-CLEANING APPARATUS; ION BEAM AND BACK-SPUTTERING EQUIPMENT (CHAMBER FOR GROUND TESTS; TEST PACKAGES FOR KC135 AND SOUNDING ROCKET). SCANNING ELECTRON MICROSCOPE WITH MICROPROBE. CONTAMINATION CONTROLLED HANDLING AND STORAGE EQUIPMENT. ENVIRONMENTAL INSTRUMENTATION, PHOTOGRAPHY.

PROCEDURE:

PRELIMINARY GROUND-BASED CHEMICAL CLEANING; APPROPRIATE SOLVENT AND ACID SCRUBS, RINSE, NITROGEN (FILTERED VIA MILLIPORE GUN) DRY, COOL (40 MINUTES). CONTAMINANT-FREE HANDLING AND STORAGE. GROUND ULTRA-CLEANING; INSTALL IN CLEANING CHAMBER, AND EVACUATE AND PERFORM BACK-SPUTTERING AND/OR ION BEAM SCRUBBING (1 HOUR). TERMINATE, REPRESSURIZE WITH PURE, DRY N_2 (10 MINUTES). CONTAMINANT-FREE HANDLING. INSTALL IN SCANNING ELECTRON BEAM MICROSCOPE AND INSPECT VIA ELECTRON SCOPE AND MICROPROBE (1 DAY). KC135 AND SOUNDING ROCKET ULTRA-CLEANING; PERFORM ALL GROUND CHEMICAL CLEANING AND HANDLING STEPS (40 MINUTES). INSTALL IN TEST PACKAGE WITH ION BEAM AND/OR BACK-SPUTTERING APPARATUS (30 MINUTES). IN FLIGHT, INITIATE EXPOSURE TO SCRUBBING AND/OR BACK-SPUTTERING (2-5 MINUTES - TOTAL FOR KC135 TRAJECTORIES, 5-10 MINUTES FOR SOUNDING ROCKET. RECORD AND COMMUNICATE DATA TO GROUND). SEAL IN CONTAMINATION SHROUD. RETURN TO GROUND. INSPECT VIA ELECTRON SCOPE AND MICROPROBE (1 DAY).

Figure III-73. Summary Definition of Requirements for Verification Experiments or Tests, 3 - Crystal Ultra-Cleaning Processes

FABRICATION OF SURFACE ACOUSTIC WAVE COMPONENTS

PURPOSE:

TO DEVELOP METHOD FOR UNIFORM DEPOSITION OF ADHERENT CONDUCTOR FILMS OF ALUMINUM AND/OR GOLD AT <1 MICRON THICKNESSES, WITH INITIAL FLASH LAYERS OF CADMIUM <100 A°. TO ASCERTAIN QUALITY OF SUCH METHOD(S).

CONDITIONS:

IN GROUND LABORATORY AND KC135; STANDARD CONDITIONS, SEALED SPUTTERING AND/OR VAPOR DEPOSITION CHAMBER (10^{-3} TO 10^{-6} N/M^2). IN SOUNDING ROCKET; AMBIENT VACUUM. IN KC135 AND SOUNDING ROCKET; ZERO "G" TRAJECTORY.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

CRYSTALS OF LITHIUM NIOBATE, SAPPHIRE, QUARTZ, OPTICALLY POLISHED ONE FACE, FROM 1 MM TO 20 CM SIZE. ULTRA-CLEANED. TOTAL <1 KG.

EQUIPMENT, APPARATUS REQUIRED:

SPUTTERING AND VAPOR DEPOSITION EQUIPMENT. VACUUM CHAMBER (FOR GROUND TESTS). TEST PACKAGES FOR KC135 AND SOUNDING ROCKET. ENVIRONMENTAL INSTRUMENTATION AND PHOTOGRAPHY. RECORDING AND TELEMETERY FOR DATA. GROUND-BASED EQUIPMENT FOR COATING ADHERENCE TESTING, ELECTRICAL CONTINUITY AND CONDUCTIVITY CHECKS. CONTAMINATION-CONTROLLED HANDLING AND STORAGE EQUIPMENT.

PROCEDURE:

IN GROUND LABORATORY, TWO HOURS OF INITIAL PUMP DOWN TIME IS GENERALLY REQUIRED. FOR KC135 AND SOUNDING ROCKET TESTS, IT IS ADVISABLE TO EXPOSE THE CRYSTALS TO VACUUM PRIOR TO TEST, TO PERMIT OUTGASSING FROM THE SURFACES. HARD VACUUM, ALLOW ONE HOUR FOR ADEQUATE OUTGASSING. STARTING WITH CLEAN CRYSTALS, SELECTED EVAPORATING AND/OR SPUTTERING EQUIPMENT, APPLY ~100A° CHROMIUM FLASH COATING (1 MINUTE), DEPOSIT THIN (500 A° TO 1500 A°) FILMS OF GOLD AND ALUMINUM OVER ONE FACE OF EACH CRYSTAL (5 MINUTES). FOR GROUND METALLIZATION TEST; CONTAMINANT-FREE HANDLING, INSTALL IN MICROSCOPE FOR INSPECTION AND MEASUREMENT. FOR KC135 AND SOUNDING ROCKET, SEAL IN CONTAMINATION SHROUD. RECOVER AND INSTALL IN MICROSCOPE FOR INSPECTION AND MEASUREMENT. MICROSCOPIC EXAMINATION (1 DAY).

Figure III-74. Summary Definition of Requirements for Verification Experiments or Tests, 4 - Surface Metallization

FABRICATION OF SURFACE ACOUSTIC WAVE COMPONENTS

PURPOSE:

TO SELECT WORKING ELECTRON AND X-RAY RESIST COATINGS, AND DEFINE PERFORMANCE OF TECHNIQUES FOR APPLYING THEM TO SUBSTRATES.

CONDITIONS:

GROUND LABORATORY; STANDARD CONDITIONS, AND ONE "G". FOR ALL TESTS; SEALED CHAMBER WITH HARD VACUUM (10^{-3} TO 10^{-6} N/M^2). IN DROP TOWER AND KC135, ZERO "G" PATH.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

CRYSTALS 1 MM TO 10 CM LONG, WITH WORKING SURFACE OPTICALLY FLAT AND CLEAN. VARIOUS ELECTRON RESISTS AND X-RAY RESISTS.

EQUIPMENT, APPARATUS REQUIRED:

VACUUM CHAMBER WITH SPINNER TABLE, STANDARD SPINNERS, MODIFIED SPINNERS. MICROSCOPE FOR VISUAL INSPECTION OF THE COATING FILM. ENVIRONMENTAL INSTRUMENTATION AND PHOTOGRAPHY. FLIGHT DATA RECORDING AND TELEMETRY.

PROCEDURE:

INSTALL SPIN TABLE IN VACUUM CHAMBER (1 HOUR). MOUNT SPINNER ON TABLE, INSTALL CRYSTAL SLAB ON SPINNER, APPLY PRECALCULATED AMOUNT OF RESIST SOLUTION, SEAL CHAMBER, EVACUATE (30 MINUTES). FOR DROP TOWER AND KC135; INSTALL REPRESSURIZE, REMOVE COATED SLAB (10 MINUTES). STORE FOR BAKE OUT AND INSPECTION. GROUND BASED INSPECTION; PLACE COATED SLAB IN MICROSCOPE, FOCUS ON SELECTED POINT (5 MINUTES). MOVE SLAB HORIZONTALLY, NOTE CHANGE IN COATING FILM THICKNESS BY VARIATION IN FOCUS (1 DAY). IN TEST OF PROTOTYPE SPINNER, FINAL EVALUATION REQUIRES LITHOGRAPHY OF A TEST PATTERN WITH A STANDARD MASK FOR AUTHORITATIVE CHECK.

Figure III-75. Summary Definition of Requirements for Verification Experiments or Tests, 5 - Application of Resist Coating

PURPOSE:

TO ASSESS VIBRATION LEVELS IN SPACECRAFT; DETERMINE IF, AN WHAT, VIBRATION CONTROLS ARE NECESSARY; AND PROVIDE HIGH RESOLUTION MASK CAPABILITY, WITH HIGHER ACCURACY THAN GROUND FACILITY, FOR S.A.W. CIRCUITRY UP TO 30 GHz RANGE. (SEE APPENDIX D, BOOK 2, VOLUME II.)

CONDITIONS:

FOR IN-ORBIT VIBRATION DATA; VARIETY OF SPACECRAFT NATURAL FREQUENCIES, VIBRATION-INDUCING OPERATIONS. GROUND LABORATORY; STANDARD CONDITIONS AND SHAKERS SIMULATING IN-ORBIT LEVELS OF VIBRATION. SPACECRAFT TEST; ZERO "G", HARD VACUUM, VIBRATION ISOLATION AS REQUIRED.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

FOR MASK FABRICATIONS; HIGH RESOLUTION, PROGRAMMABLE ELECTRON BEAM (FROM TEST 1), MASK MATERIALS. FOR GROUND LABORATORY TESTS; VARIOUS VIBRATION ISOLATORS. FOR EXPOSURE TESTS; MASKS FABRICATED ABOVE, AND LABORATORY CONTROL SAMPLE MASKS FABRICATED ON GROUND.

EQUIPMENT, APPARATUS REQUIRED:

FOR VIBRATION INFORMATION; VARIETY OF SPACECRAFT WITH VARIOUS OPERATING EQUIPMENT, INSTRUMENTED WITH STRAIN GAUGE SEISMOMETERS AND/OR MILLI-G METERS. RECORDERS AND TELEMETRY. FOR MASK FABRICATION TESTS; GROUND-BASED VIBRATION SIMULATORS, SUBSTRATE HOLDING FIXTURE, CONTAMINATION CONTROL. VACUUM CHAMBER (GROUND LABORATORY TESTS; TEST PACKAGE, INCLUDING AUTOMATION EQUIPMENT (SPACECRAFT TESTS). MASK ACCURACY TESTING VIA SCANNING ELECTRON MICROSCOPE. FOR EXPOSURE TESTING; SCANNING (SOFT) X-RAY MACHINE USING ALLUMINUM TARGET.

PROCEDURE:

(A) IN-ORBIT VIBRATION INFORMATION; ANALYSIS OF EXISTING DATA (6 MONTHS), INSTRUMENTATION OF EARLY PLANNED SPACECRAFT OF VARIOUS VIBRATION CHARACTERISTICS AND ANALYSIS OF RESULTS (1 TO 5 YEARS). VIBRATION MEASUREMENTS (1 DAY PER RUN.)

(B) GROUND LABORATORY TESTS OF ISOLATION METHODS AND EFFECTS ON ELECTRON BEAM WRITING; INSTALL PREPROGRAMMED ELECTRON BEAM GUN ON SIMULATOR WITH VARIOUS ISOLATORS AND DYNAMICALLY BALANCE (1 DAY TO 1 WEEK). INSTALL MASK HOLDING FIXTURE. INSTALL MASK BLANK IN FIXTURE AND ALIGN BEAM (½ HOUR). INITIATE SIMULATED SPACECRAFT VIBRATIONS AND "WRITE" MASK (1 TO 40 HOURS). TERMINATE TEST, REMOVE MASK AND MEASURE ACCURACY VIA SCANNING ELECTRON BEAM MICROSCOPE (1 DAY).

Figure III-76A. Summary Definition of Requirements for Verification Experiments or Tests, 6 - Mask Fabrication

PROCEDURE: (CONTINUED)

(C) SPACECRAFT TEST OF ELECTRON BEAM MASK FABRICATION; ASSEMBLE ELECTRON BEAM GUN, MASK HOLDING FIXTURE, MASK BLANKS, PROGRAMMER INTO TEST PACKAGE AND CHECKOUT (1 WEEK TO 1 MONTH). MAINTAIN CONTAMINATION CONTROL AND PROTECTIVE HANDLING. INSTALL TEST PACKAGE INTO SPACECRAFT AND CHECKOUT (1 TO 2 WEEKS). AFTER ACHIEVING ORBIT, AUTOMATICALLY CUT MASKS (1 TO 40 HOURS EACH). TERMINATE. RECOVER MASKS VIA SHUTTLE. RETURN TO LABORATORY FOR ACCURACY MEASUREMENT.

(D) SPACECRAFT CHECK OF MASK FIDELITY AND ACCURACY (PART OF TEST 7). AUTOMATICALLY ALIGN NEWLY-CUT MASK WITH RESIST-COATED SUBSTRATE AND EXPOSE TO X-RAY (10 MINUTES). SIMULTANEOUSLY EXPOSE PRE-ALIGNED, PRE-CUT LABORATORY CONTROL MASK WITH SUBSTRATE AND EXPOSE. TERMINATE. RECOVER MASKS AND EXPOSED SLABS WITH SHUTTLE. RETURN TO LABORATORY FOR DEVELOPING, ETCHING, MEASUREMENT AND TESTING (1 WEEK).

(E) SPACECRAFT TEST OF ELECTRODE "WRITING" ON SUBSTRATE VIA PROGRAMMED ELECTRON BEAM (PART OF TEST 7); PROCEDURE AS IN (C), USING RESIST-COATED SUBSTRATES IN-PLACE OF MASK BLANKS.

Figure III-76B. Summary Definition of Requirements for Verification Experiments of Tests, 6 - Mask Fabrication (Continued)

PURPOSE:

TO PROVIDE DATA ON EFFECTS OF VIBRATION ON EXPOSURE THROUGH MEASUREMENT OF FIDELITY AND ACCURACY OF ELECTRODE PATTERNS PRODUCED UNDER VARIOUS VIBRATION CONDITIONS TO OPTIMIZE HIGH RESOLUTION EXPOSURE METHOD FOR USE IN SPACE PROCESSING. TO SUPPORT TEST 6 IN SPACECRAFT.

CONDITIONS:

GROUND LABORATORY CONDITIONS, VARIOUS VIBRATION ISOLATION LEVELS (TROM TEST 6). SPACECRAFT CONDITIONS IN TEST 6.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

CRYSTALS (1 MM TO 10 CM) WITH CLEAN, THIN RESIST COATINGS. BEST AVAILABLE RESOLUTION LABORATORY CONTROL MASKS ($\backsim$200 A^{o} FINGERS, IF POSSIBLE).

EQUIPMENT, APPARATUS REQUIRED:

SOFT X-RAY SOURCE, CRYSTAL HOLDER, MASK ALIGNMENT JIGS. FOR SPACECRAFT TEST, SEE TEST 6. FOR MEASUREMENT AND EVALUATION (IN GROUND LABORATORY), THIN FILM PROCESSING EQUIPMENT USED FOR LITHOGRAPHY, ELECTRON BEAM MICROSCOPE, AND ELECTRONIC SIGNAL GENERATION AND RECEIVING EQUIPMENT (UP TO 30 GHz FOR EVALUATING PERFORMANCE OF SURFACE WAVE DEVICES.

PROCEDURE:

IN GROUND LABORATORY WITH SELECTED VIBRATION ISOLATION EQUIPMENT, ASSEMBLE AND ALIGN JIGS AND X-RAY SOURCE TO BE USED (1 DAY TO 1 WEEK). INSTALL AND ALIGN MASK AND SUBSTRATE IN JIG, AND EXPOSE TO X-RAY (10 MINUTES). RETRIEVE EXPOSED SUBSTRATE, DEVELOP, ETCH, CLEAN (1 HOUR). MEASURE ACCURACY WITH ELECTRON BEAM MICROSCOPE. TEST FOR ELECTRONIC PERFORMANCE (WAVE FORM, SIGNAL TO NOISE, ATTENUATION, SPECIFIC COMPONENT FUNCTIONS) (1 WEEK).

SPACE TESTS IN ZERO "G", LOW VIBRATION ENVIRONMENT; SHOWN IN TEST 6. COMPONENT FINISHING, MEASUREMENT AND TEST, AS ABOVE.

Figure III-77. Summary Definition of Requirements for Verification Experiments or Tests, 7 - Resist Exposure Tests

PROCEDURE:

TO DEMONSTRATE PROCESS EQUIPMENT PERFORMANCE, AND DEVELOP AUTOMATION CONTROL.

CONDITIONS:

ORBITAL; ZERO "G", HARD VACUUM, ZERO VIBRATION.

SPECIMENS, SAMPLES, ITEMS TO BE TESTED:

SELECTED CRYSTAL MATERIALS, CRYSTAL GROWING APPARATUS (IF SUCCESSFUL), PRE-LITHOGRAPHIC STEPS (IF FEASIBLE), MASK FABRICATION EQUIPMENT, EXPOSURE EQUIPMENT.

EQUIPMENT, APPARATUS REQUIRED:

PROCESS MONITORING AND EVALUATION INSTRUMENTATION, MANUAL OVERRIDE AND ADJUSTMENT CONTROLS AND DISPLAYS, COMMUNICATIONS. GROUND LABORATORY FOR FINISHING EVALUATION AND TEST OF PRODUCTS, AS IN TEST 7.

PROCEDURE:

GROW CRYSTAL, BASED ON TEST SERIES 2 (1 DAY TO 1 WEEK). ORIENT CRYSTAL (1 HOUR). CUT AND POLISH CRYSTAL, IF REQUIRED (2 TO 3 DAYS). POLE (6 HOURS). TIME FOR POLING IS ACTUALLY THAT REQUIRED FOR GRADUAL COOLING, SINCE THE CRYSTAL TENDS TO FRACTURE UNDER ABRUPT LARGE CHANGES IN TEMPERATURE. CLEAN CRYSTAL (1 HOUR). METALLIZE SURFACE (5 TO 10 MINUTES FOR ACTUAL METALLIZATION). LONGER TIMES REQUIRED IN GROUND FACILITIES, DUE TO TIME REQUIRED FOR PUMP DOWN AND DEGASSING. IN SPACE VACUUM PROBABLY 1 HOUR IS ADEQUATE. APPLY RESIST (1 MINUTE). BAKE OUT FOR ONE HOUR. MASK ALIGNMENT AND EXPOSURE EFFECTIVELY A SINGLE STEP (10 MINUTES). SEAL AGAINST CONTAMINATION ($\sim$1 MINUTE). PACKAGE FOR RETURN. IN GROUND LABORATORY, CONTAMINATION CONTROLLED HANDLING, DEVELOP, ETCH, AND CLEAN (1 HOUR). MEASURE AND TEST AS IN TEST SERIES 7.

Figure III-78. Summary Definition of Requirements for Verification Experiments or Tests, 8 - Prototype Test of Overall Process

KNOWLEDGE GAPS	EXPERIMENTS AND VERIFICATION TESTS	EXPERIMENT AND TEST REQUIREMENTS (SUMMARY)
CRYSTAL GROWTH MATERIALS AND METHOD		
BEST FOR: LARGEST SIZE HIGHEST QUALITY FREEDOM FROM DISLOCATIONS SURFACE/PRINCIPAL PLANE ALIGNMENT COST	EXPERIMENTS (EXTENSION OF PRESENT GROUND AND SKYLAB TECHNIQUES) OF FLUX GROWTH, CZOCHRALSKI, OTHER GROWTH, OF LITHIUM NIOBATE, SAPPHIRE, SPINEL, LITHIUM TANTALATE, BISMUTH GERMANATE FOR SELECTION OF METHOD AND MATERIALS (LIMITS OF SIZE, PURITY, FREEDOM FROM DISLOCATIONS, ALSO SURFACE/PLANE ALIGNMENT)	STANDARD GROUND LAB WITH STANDARD FLUX GROWTH, CZOCHRALSKI, OTHER CRYSTAL GROWING EQUIPMENT, CANDIDATE CRYSTAL MATERIALS RIGOROUS THERMAL, VIBRATION, ATMOSPHERE CONTROL AND INSTRUMENTATION; PHOTOGRAPHIC MEASUREMENT APPARATUS ~ 2 DAYS PER RUN, MANUAL CONTROL LATER, ORBITING SPACE FACILITY WITH SPACE CRYSTAL GROWING APPARATUS, SELECTED MATERIALS, RIGOROUS ENVIRONMENTAL CONTROL AND INSTRUMENTATION, PHOTOGRAPHIC APPARATUS. ~ 2 DAYS PER RUN, MANUAL CONTROL, TELEMETRY OF DATA, RECOVERY OF APPARATUS AND PRODUCED CRYSTALS, CRYSTAL MEASUREMENT AND EVALUATION IN GROUND LAB.
ACOUSTIC SURFACE ULTRACLEANING METHOD*		
ION BEAM SCRUBBING IN HARD VACUUM – COST, WEIGHT, QUALITY OF RESULTS BACK SPUTTERING IN ZERO "G" HARD VACUUM – TECHNOLOGY, COST, WEIGHT, QUALITY OF RESULTS	TESTS TO ESTABLISH PROCEDURE, PERFORMANCE, CONTAMINATION EFFECTS OF SPACE METHODS (IF REQUIRED), COMPARISON OF ION BEAM SCRUBBING VS BACK SPUTTERING	ZERO "G" AIRCRAFT AND SOUNDING ROCKET TESTS, ION BEAM SCRUBBING AND BACK SPUTTERING APPARATUS, ENVIRONMENT INSTRUMENTATION, PHOTOGRAPHY, RIGOROUS CONTAMINATION CONTROL, SAMPLE CRYSTAL SLABS, CLEANING CHAMBER, AUTOMATED, 20 SEC TO 10 MIN. PER RUN, RECORDING AND/OR TELEMETRY OF DATA, RECOVERY OF SAMPLE SLABS, CLEANING CHAMBER, PHOTOGRAPHY, TESTS REQUIRED ONLY IF PREFERRED IN-SPACE ULTRA-CLEANING IS ADOPTED MODE.
SURFACE METALLIZATION METHOD*		
SPUTTERING IN ZERO "G" HARD VACUUM – TECHNOLOGY, COST, WEIGHT, QUALITY OF RESULTS VAPOR DEPOSITION IN ZERO "G", HARD VACUUM – TECHNOLOGY, COST, WEIGHT, QUALITY OF RESULTS	TESTS TO ESTABLISH PROCEDURE, PERFORMANCE, CONTAMINATION OF SPACE METHODS (IF REQUIRED), COMPARISON OF SPUTTERING VS. VAPOR DEPOSITION.	DROP TOWER AND ZERO "G" AIRCRAFT TESTS, SPUTTERING AND VAPOR DEPOSITION APPARATUS, ENVIRONMENT INSTRUMENTATION, PHOTOGRAPHY, RIGOROUS CONTAMINATION CONTROL, SAMPLE CRYSTAL SLABS, METALLIZATION CHAMBER, AUTOMATED, 4 SEC TO 20 SEC PER RUN, RECORDING AND/OR TELEMETRY OF DATA, RECOVERY OF SAMPLE SLABS, METALLIZATION CHAMBER, PHOTOGRAPHY. TESTS REQUIRED ONLY IF PREFERRED IN-SPACE METALLIZATION IS ADOPTED MODE.
METHOD OF APPLICATION OF RESIST COATING*		
METHOD FOR ZERO "G", TECHNOLOGY COST, WEIGHT, QUALITY OF RESULTS	TEST TO EVALUATE (POSSIBLY MODIFY) "SPINNER" METHOD FOR PROCEDURE, PERFORMANCE, CONTAMINATION EFFECTS.	ZERO "G" AIRCRAFT AND SOUNDING ROCKET TESTS, "SPINNER" AND MODIFIED "SPINNER" APPARATUS, ENVIRONMENT INSTRUMENTATION, PHOTOGRAPHY, RIGOROUS CONTAMINATION CONTROL, SAMPLE CRYSTAL SLABS, RESIST-APPLICATION CHAMBER, AUTOMATED, 20 SEC TO 10 MIN. PER RUN, RECORDING AND/OR TELEMETRY OF DATA, RECOVERY OF SAMPLE SLABS, RESIST-APPLICATION CHAMBER, PHOTOGRAPHY. TESTS REQUIRED ONLY IF PREFERRED IN-SPACE RESIST-COATING IS ADOPTED MODE

Figure III-79A. Definition of Requirements for Experiments to Verify Selected Approach for Fabrication of Surface Acoustic Wave Components

KNOWLEDGE GAPS	EXPERIMENTS AND VERIFICATION TESTS	EXPERIMENT AND TEST REQUIREMENTS (SUMMARY)
EFFECTS OF ORBITAL CONDITIONS ON MASK FABRICATION*		
VIBRATION LEVELS IN SPACECRAFT, ISOLATION, FABRICATION METHODS TO ACHIEVE 200 Å LINES AND SPACES	EXPERIMENTS TO MEASURE (GROUND, AUTOMATED SPACECRAFT, MANNED SPACECRAFT) VIBRATION LEVELS, EFFECTS ON MASK FIDELITY AND ACCURACY. TESTS TO DEVELOP SPACE MASK FABRICATION METHOD, EVALUATE PROCESS (INCLUDING VIBRATION ISOLATION)	GROUND AND SPACECRAFT EXPERIMENTS, VIBRATION-MEASURING INSTRUMENTATION. AUTOMATED. ONE DAY PER RUN. TELEMETRY OF DATA. LATER GROUND AND SPACECRAFT TESTS OF MASK FABRICATION APPARATUS, CANDIDATE VIBRATION ISOLATORS, MASK SAMPLES. VIBRATION INSTRUMENTATION. AUTOMATED. ONE DAY PER RUN. TELEMETRY OF DATA, RECOVERY OF FABRICATED MASKS, FABRICATION APPARATUS, GROUND LAB WITH SOFT X-RAY LITHOGRAPHY SYSTEM, VIBRATION ISOLATION, VIBRATION MEASURING INSTRUMENTATION, SAMPLE MASKS, RESIST-COATED SUBSTRATES, AUTOMATED. ~ MINUTE PER RUN.
EFFECTS OF ORBITAL CONDITIONS ON EXPOSURE *		
VIBRATION LEVELS IN SPACECRAFT, ISOLATION METHODS TO ACHIEVE 200 Å LINES AND SPACES. SHIELDING. TECHNOLOGY, COST, WEIGHT, QUALITY OF RESULTS TRADE-OFFS WITH PROGRAMMED ELECTRON BEAM	EXPERIMENTS TO MEASURE VIBRATION LEVELS (AS ABOVE), EFFECTS ON EXPOSURE FIDELITY AND ACCURACY. TESTS TO DEVELOP EXPOSURE METHODS, EVALUATE PROCESS INCLUDING VIBRATION ISOLATION, COMPARISON OF X-RAY LITHOGRAPHYVS PROGRAMMED ELECTRON BEAM.	TESTS OF EXPOSURE SYSTEM. RESIST-COATED CRYSTAL SLABS, VARIOUS FIDELITY MASKS. X-RAY SOURCE. SELECTED VIBRATION ISOLATORS. VIBRATION-MEASURING INSTRUMENTATION. PROGRAMMED ELECTRON BEAM. AUTOMATED WITH MANUAL CONTROL. MINUTE PER RUN. TELEMETRY OF DATA, RECOVERY OF EXPOSED SLABS, MASKS, ELECTRON BEAM, APPARATUS. TESTS REQUIRED ONLY IF PREFERRED IN-SPACE EXPOSURE IS ADOPTED MODE.
PERFORMANCE OF OVERALL PROCESS*		
TIMING, STEPS CAPABLE OF/FEASIBLE FOR, AUTOMATION	TESTS AND DEMONSTRATION OF PROTOTYPE EQUIPMENT, SYSTEM, AND PRODUCTS.	SHUTTLE FACILITY TESTS AND DEMONSTRATION. FIRST, MAJOR EQUIPMENTS; LATER, TOTAL SYSTEM. PROCESS INSTRUMENTATION, SAMPLE RAW MATERIALS. FIRST, MANUAL; LATER AUTOMATED WITH MANUAL CONTROL. UP TO 2 DAYS PER RUN. TELEMETRY OF DATA. RECOVERY OF INTERMEDIATE AND FINAL PRODUCTS, ALL APPARATUS. STANDARD S.A.W. GROUND TEST LAB. ELECTRONIC EQUIPMENT TEST EQUIPMENT FOR S.A.W. PERFORMANCE.

* IF DECISION IS FOR PREFERRED IN-SPACE PROCESS STEP

Figure III-79B. Definition of Requirements for Experiments to Verify Selected Approach for Fabrication of Surface Acoustic Wave Components

III.9 MISSION PROFILES FOR EXPERIMENTS AND TESTS

The preceding sections have, in the process of defining experiment and test requirements, identified specific types of facilities in which such experiments and tests should be carried out.

This section provides an insight into operating conditions and timing involved in utilizing such facilities to meet conditions and procedures documented as experiment and test requirements.

Review of the Experiment and Test Requirements, Sections III.5 through III.8, reveals the need for various ground laboratories, drop tower, KC-135 "Zero G" aircraft, sounding rockets, spacecraft, and Space Shuttle, as experiment and test facilities. This section will first extract the procedures data from the preceding sections to summarize the experiment and test mission profiles, and then review pertinent mission profile data for these facilities

III.9.1 MISSION PROFILES FOR EXPERIMENTS AND TESTS IN SUPPORT OF SEPARATION OF ISOENZYMES

The experiments and tests required to develop this process rely heavily on ground laboratory work for both study of the phenomenology involved and developmental work on processing equipment. In general, preliminary ground laboratory experiments form the basis for subsequent verification testing in spacecraft. As an example, Figure III-80, which provides mission profile data on all the Isoenzyme Experiments and Tests, shows that the ground laboratory tests of enzyme mobility vs voltage gradient provide the background data for the zero-g verification testing in the automated spacecraft.

The "durations" listed for the functions pre-purification and bio-assay, 2 months and 1 day, respectively, are indicative of the fact that some portions of the process "separation of isoenzymes" are planned for ground operations rather than orbital.

In fact, only the actual separation process step requires orbital operations, and the experiments and tests of other steps are performed only to assure that the separation equipment, specimens and sample products are not impaired by other process steps.

Thus, for example, tests IIIB and IIIA call for centrifuge simulation of Shuttle launch loads on large pore gels and separation equipment in order to determine whether ground-formed gels and standard separation equipment, respectively, will survive launch to orbit, where separation will be performed.

The place of the profiles presented in Figure III-80 within the context of facilities mission profiles can be observed by perusing the facility profiles given in Section III.9.5.

EXPERIMENT TEST	DURATION	SEQUENCE OF FUNCTIONS	EXPERIMENT/TEST CONDITIONS	CREW TASKS
GROUND LAB EXPERIMENTS/TESTS				
1A ENZYME MOBILITY VS VOLTAGE GRADIENT	1-2 DAYS RUN (15-20 RUNS REQ'D)	SET UP, PREP OF SPECIMENS, SEPARATION, REMOVAL OF GELS, STAINING, ANALYSIS	GROUND LAB	SET UP, PREP, INSERT SPECIMENS, INITIATE SEPARATION, OBSERVATION, TERMINATE SEPARATION, REMOVE GELS, STAIN, ANALYZE
1B EFFECTS OF HEATING AND CONVECTION	2 DAYS/RUN (10 RUNS REQ'D)	SET UP, PREP OF SPECIMENS, SEPARATION, THERMAL LOAD, REMOVAL OF GELS, STAINING, BAND DISTORTION MEASUREMENT	GROUND LAB, LOCAL HEATING AND COOLING (± 30°C)	SET UP, PREP, INSERT SPECIMENS, INITIATE SEPARATION, OBSERVATION, INITIATE AND CONTROL HEATING/COOLING, REMOVE GELS, STAIN, MEASURE BAND DISTORTION.
1C EFFECTS OF PATH LENGTH ON ELECTROPHORESIS	1 2 DAYS RUN (10-15 RUNS REQ'D)	AS IN 1A	GROUND LAB	AS IN 1A
1D EFFECTS OF PATH LENGTH ON ISOELECTRIC FOCUSSING	1 1 2-2 1/2 DAYS RUN (10-15 RUNS REQ'D)	AS IN 1A, USING ISOELECTRIC FOCUSSING MATERIALS AND APPARATUS	GROUND LAB	AS IN 1A
IIA SELECTION OF SEPARATION SYSTEM	PRE-PURIFICATION, 2 MOS, TESTS, 1-2 DAYS/RUN (15-30 RUNS REQ'D)	PRE-PURIFICATION, BIO-ASSAY, SEPARATION, STAINING, ANALYSIS, STORAGE, SET UP, PREP OF SPECIMENS, SEPARATION (VARIATION OF BUFFERS, GELS, ETC.), REMOVAL OF GELS, STAINING, ANALYSIS	GROUND LAB	PRE-PURIFICATION, ASSAY, INITIATE SEPARATION, TERMINATE SEPARATION, REMOVE SPECIMENS, STAIN, ANALYZE, STORE, PROCEED AS IN 1A. MODIFY BUFFERS, GELS, ETC. AS REQUIRED.
IIB DEMONSTRATION OF PREPARATIVE-SCALE SEPARATION	3-4 DAYS RUN (3-5 RUNS REQ'D)	AS IN IIA WITHOUT VARIATIONS	GROUND LAB	AS IN IIA WITHOUT MODIFICATIONS
IIIA LAUNCH ENVIRONMENT TESTS ON STANDARD SEPARATION EQUIPMENT	10 MIN/RUN (5-10 RUNS REQ'D)	SET UP, INITIATE TRAJECTORY AND LOADING SIMULATION, RECORD DATA, TERMINATE	SIMULATED SHUTTLE PAYLOAD BAY VIBRATION, SHOCK ACCELERATION, THERMAL, ATMOSPHERE	PREPROGRAM CENTRIFUGE, MOUNT EQUIPMENT IN GONDOLA, CHECKOUT, INITIATE RECORDING AND TEST, TERMINATE, REMOVE EQUIPMENT.
IIIB LAUNCH ENVIRONMENT TESTS ON GELS	AS IN IIIA	AS IN IIIA	AS IN IIIA	AS IN IIIA
IIIC STORAGE AND RECONSTITUTION OF SPECIMENS	PRE-PURIFICATION, 2 MOS., STORAGE, 8 WEEKS-BIO-ASSAY, 1 DAY/SAMPLE	PRE-PURIFICATION, TREAT FOR STORAGE, PACKAGE, STORE, SAMPLE (ONCE PER WEEK), BIO-ASSAY	CONTROLLED LAB	PRE-PURIFICATION, PROCESSING, PACKAGING, SAMPLING, BIO-ASSAY.

Figure III-80A. Summary Mission Profiles for Experiments in Support Of Separation of Isoenzymes

	EXPERIMENT TEST	DURATION	SEQUENCE OF FUNCTIONS	EXPERIMENT TEST CONDITIONS	CREW TASKS
IIID	POST SEPARATION STORAGE AND HANDLING OF PRODUCTS	STORAGE, 8 WEEKS BIO-ASSAY, 1 DAY/ SAMPLE	TREAT FOR STORAGE, PACKAGE, STORE, SAMPLE (ONCE PER WEEK) BIO-ASSAY	CONTROLLED LAB	PROCESSING, PACKAGING, SAMPLING, BIO-ASSAY
IVA, B,C	DESIGN TESTING OF SPACE-BORNE PROCESSING, EQUIPMENT AND SUPPORT	AS IN IIIA	AS IN IIB	AS IN IIB	AS IN IIB
	ZERO "G" AIRCRAFT AND SOUNDING ROCKET EXPERIMENTS AND TESTS				
IVA' B',C'	DESIGN TESTING (ZERO "G" AIRCRAFT)	20-40 SEC/TEST (20-40 TESTS REQ'D)	CARRY ON TEST PACKAGE, TAKE OFF, INITIATE TRAJECTORY, INITIATE TEST, RECORD, OBSERVE, TERMINATE, TEST, REPEAT.	INTERNAL TO AIRCRAFT 20-40 SEC ZERO "G"	PREFLIGHT PREP, POSITIONING OF TEST PACKAGE DURING MANEUVER, INITIATE TEST, CONTROL PACKAGE POSITION, RETRIEVE PACKAGE, TERMINATE TEST
	AUTOMATED SPACECRAFT TESTS (DESIRED, BUT COULD DELAY UNTIL SHUTTLE AND DELAY PROGRAM)				
1A'	ENZYME MOBILITY VS VOLTAGE GRADIENT	1DAY/RUN (5-10 RUNS REQ'D)	PRELAUNCH OPERATIONS, MONITOR, INITIATE TEST, TELEMETRY DATA, TERMINATE TEST, REPEAT, (STORED SPECIMENS AND RECOVERY DESIRED)	PRESSURIZED SPACECRAFT, LONG TERM ZERO "G".	PRELAUNCH OPERATIONS, TEST MONITORING AND CONTROL, RECOVERY, POST-FLIGHT AS IN 1A.
1B'	EFFECTS OF HEATING	1DAY/RUN (5-10 RUNS REQ'D)	AS IN 1A'	AS IN 1A'	AS IN 1A', POSTFLIGHT AS IN 1B.
1C'	EFFECTS OF PATH LENGTH ON ELECTROPHORESIS	1-2 DAYS/RUN (5-10 RUNS REQ'D)	AS IN 1A'	AS IN 1A'	AS IN 1A'
1D'	EFFECTS OF PATH LENGTH ON ISOELECTRIC FOCUSSING	1-2 DAYS/RUN (5-10 RUNS REQ'D)	AS IN 1A'	AS IN 1A'	AS IN 1A'
IVA' B'C'	DESIGN TESTING	1 DAY/RUN (5-10 RUNS REQ'D)	AS IN 1A'	AS IN 1A'	AS IN 1A'
	SHUTTLE SORTIE LAB TESTS				
IVA'' B'',C''	DESIGN TESTING	UP TO 2 DAYS/RUN (5-10 RUNS REQ'D)	PRELAUNCH, LAUNCH, ON-ORBIT SET UP, INITIATE TEST, RECORD AND TELEMETRY DATA, TERMINATE TEST, STOW, DEORBIT, RECOVER.	PRESSURIZED SORTIE LAB ENVIRONMENT. LONG TERM ZERO "G"	CHECKOUT, INITIATE, MONITOR, CONTROL, MAINTAIN, REPAIR, TERMINATE TEST, STOW GEAR.
V	PROTOTYPE DEMONSTRATION	1-2 DAYS/RUN (2-3 RUNS REQ'D)	AS IN IVA'', B'', C''	AS IN IVA'', B'', C''	AS IN IVA'', B'', C''

Figure III-80B. Summary Mission Profiles for Experiments in Support Of Separation of Isoenzymes

III.9.2 MISSION PROFILES FOR EXPERIMENTS AND TESTS IN SUPPORT OF PROCESSING TRANSPARENT OXIDES

The considerable amount of earlier analytical and experimental work that characterize this product area has been directed at other means of avoiding devitrification than Zero "G" processing. Thus, the experiments and tests required are aimed at providing some small amount of material data, but, mainly, are aimed at establishing free suspension process feasibility.

Ground laboratory experiments and tests require rather long running times, since most of them measure results in terms of characterization of the final product. and that is a time-consuming task. In addition, the cooling function, in the ground tests will be performed slowly to minimize convection effects (in order to minimize nucleation sites). That function is also time-consuming.

The "duration" and "functions" columns of Figure III-81 note such functions, and the shorter durations of functions during tests in the drop tower, KC-135 and sounding rockets. Such short durations could not be expected to provide data for materials characterization. Rather these tests, are aimed at assessing the capability of various techniques and equipments to carry on specific processing functions, such as positioning, quenching, etc. The short duration tests result in measurements which provide means of extrapolating performance to nominal process durations.

As noted earlier, these experiment and test profiles "fit" into the test portion of overall facility mission profiles discussed in Section III.9.5.

III.9.3 MISSION PROFILES FOR EXPERIMENTS AND TESTS IN SUPPORT OF FABRICATING TUNGSTEN X-RAY TARGETS

The possibility of a soon-to-be realized improvement in x-ray target life through free suspension processing has provided considerable impetus to this program. The Ground Laboratory experiments and tests included in Figure III-82 have already been

EXPERIMENT TEST	DURATION	SEQUENCE OF FUNCTIONS	EXPERIMENT TEST CONDITIONS	CREW TASKS
GROUND LAB EXPERIMENTS AND TESTS				
(1) SELECTION OF INITIAL OXIDES	1–2 DAYS/RUN (6–12 RUNS REQ'D)	ANALYSIS OF SAMPLES, MELT, GAS ANALYSIS, COOL, X–RAY CRYSTALLOGRAPHY.	GROUND LAB, ~3000°C FURNACE, INERT GAS, VACUUM TO 1 ATM.	SAMPLE PREP, SAMPLE ANALYSIS, LOAD FURNACE, INITIATE INSTRUMENTATION AND TEST, MONITOR AND CONTROL, X–RAY CRYSTALLOGRAPHY.
(2) DEFINITION OF PROCESSING CONDITIONS	2 HRS/RUN (6–12 RUNS REQ'D)	PREP OF SPECIMENS, HEAT, MELT, VARY TEMP., GAS, PRESSURE, MATERIAL CHARACTERIZATION.	GROUND LAB, ~3000°C FURNACE INERT GAS, VARYING GAS, PRESSURES, TEMP.	SPECIMEN PREP, SET UP TEMP., GAS, PRESSURE CONDITIONS, INITIATE TEST, MONITOR AND CONTROL, MATERIAL CHARACTERIZATION.
(3) HEATING, MELTING METHODS AND DATA	8 HRS/RUN (6–12 RUNS REQ'D)	PREP,, HEAT, MELT, COOL, MATERIAL CHARACTERIZATION.	GROUND LAB, TEMP/TIME VARIATION TO ~3000°C, INERT GAS PRESS. FROM VACUUM TO 1 ATM., COOL.	SPECIMEN PREP, INITIATE TEST, MONITOR AND CONTROL MATERIAL CHARACTERIZATION.
(4) POSITIONING METHODS AND DATA	1 DAY/RUN (4 RUNS REQ'D)	SET UP POSITIONING APPARATUS, LEVITATE AND POSITION, REPEAT FOR VARIOUS SPECIMEN WEIGHTS AND POSITIONING METHODS.	GROUND LAB, LEVITATION CHAMBER, GAS/VACUUM AS REQUIRED FOR POSITIONING TECHNIQUE.	SET UP, INITIATE, POSITIONING MONITOR AND CONTROL POSITIONING APPARATUS.
(5) INITIAL PROCESS FEASIBILITY	7–9 HRS/RUN (6–12 RUNS REQ'D)	SPECIMEN PREP, PRE–TEST MATERIALS CHARACTERIZATION, TEST PREP, LEVITATION, HEATING, MELT, SUPERHEAT, SUPERCOOL AND COOL, MATERIALS CHARACTERIZATION.	GROUND LAB WITH LEVITATION CHAMBER FURNACE ~3000°C, INERT GAS (VACUUM TO 1 ATM), SELECTED HEATING PROFILE, COOLING PROFILE, MATERIALS CHARACTERIZATION.	SPECIMEN PREP, MATERIALS CHARACTERIZATION (PRE– AND POST TEST), TEST EQUIP. SETUP, TEST INITIATION, MONITOR AND CONTROL.
(6) SHAPE FORMING METHODS AND DATA	7–9 HRS/RUN (3–6 RUNS REQ'D)	AS IN (5), PLUS FORMING DURING MELT, SUPERHEAT, SUPERCOOL, COOL.	AS IN (5), PLUS ACCOMMODATION FOR ALTERNATIVE FORMING APPARATUS.	AS IN (5)

Figure 81A. Summary Mission Profiles for Experiments in Support Of Transparent Oxide Processing

EXPERIMENT TEST	DURATION	SEQUENCE OF FUNCTIONS	EXPERIMENT TEST CONDITIONS	CREW TASKS
(7) EQUIPMENT DESIGN DATA ACQUISITION, ENGINEERING DEVELOPMENT, QUALIFICATION	1/2 HR–5 DAYS/ RUN	VARY WITH COMPONENT, SUB–SYSTEM TESTED AND SPECIFIC TEST.	VARIED	SETUP, CHECKOUT, MONITOR, CONTROL, TERMINATE TEST.
DROP TOWER, KC135, AND SOUNDING ROCKET EXPERIMENTS AND TESTS				
(4A) POSITIONING METHODS AND DATA	DROP TOWER, 3–10 SEC (4–6 RUNS REQ'D) KC135, 20–40 SEC, (10–20 RUNS REQ'D) SOUNDING ROCKET, 6–10 MIN, (2 RUNS REQ'D)	LOAD TEST PACKAGE, INITIATE MISSIONS, INITIATE TEST AT START OF ZERO "G", RECORD POSITION, VELOCITY, ACCELERATION DATA, TERMINATE TEST RECOVER PACKAGE.	~ ZERO "G"	LOAD PACKAGE, INITIATE MISSION, (FOR KC135, MONITOR AND CONTROL PACKAGE) RECOVER PACKAGE.
(5A) INITIAL PROCESS FEASIBILITY	6–10 MIN.	AS IN (4A), PLUS RECORD PROCESS INSTRUMENTATION DATA	~ ZERO "G"	AS IN (4A)
SHUTTLE–BASED EXPERIMENTS AND TESTS				
(6A) SHAPE FORMING METHODS AND DATA	7–9 HRS/RUN (4–8 RUNS REQ'D)	PRELAUNCH, LAUNCH OPERATIONS, INSERT SPECIMEN ACTIVATE PROCESSING EQUIPMENT, ACTIVATE FORMING APPARATUS, COOL COLLECT FORMED SPECIMEN, REPEAT FOR EACH FORMING TECHNIQUE.	~ ZERO "G"	PRE–LAUNCH, PRE–OPERATION CHECKOUTS, LOAD SPECIMEN, TEST ACTIVATION, MONITORING, CONTROL, TERMINATION, RETRIEVE, PACKAGE SPECIMEN, CONFIGURE FOR NEXT FORMING TECHNIQUE, RECYCLE.
(7A) EQUIPMENT DESIGN DATA ACQUISITION, ENGINEERING DEVELOPMENT, QUALIFICATION	1/2 HR–5 DAYS/ RUN	VARIED	VARIED, MAINLY ZERO "G"	AS IN (7)
(8) PROTOTYPE TEST	5 DAYS	PRE–LAUNCH, LAUNCH OPERATIONS, ACTIVATE PROCESS, TERMINATE PROCESS.	ZERO "G", OPERATIONAL CONDITIONS	PRE–LAUNCH, PRE–OPERATIONAL CHECKOUTS, ACTIVATE PROCESS, MONITOR, TERMINATE PROCESSING.

Figure 81B. Summary Mission Profiles for Experiments in Support Of Transparent Oxide Processing

EXPERIMENT/TEST	DURATION	SEQUENCE OF FUNCTIONS	EXPERIMENT/TEST CONDITIONS	CREW TASKS
GROUND LAB EXPERIMENTS AND TESTS				
① VACUUM DEGASSING DATA	2–1/2 WEEKS/RUN 3–6 RUNS REQ'D)	PRE–TEST ANALYSES, SPECIMEN PREP, TEST SET UP, INITIATE HEATING, MONITOR CONDITIONS AND PRODUCTS, COOL COLLECT SPECIMENS, ANALYSES,	GROUND LAB, VACUUM CHAMBER 10^{-3} TO 10^{-6} N/M^2 FURNACE 2000 TO 2700°K	PRE– AND POST–TEST ANALYSES, SPECIMEN PREP, SET UP TEST EQUIPMENT, INITIATE TEST, MONITOR CONTROL, TERMINATE TEST, COLLECT PRODUCTS.
② SOLID TUNGSTEN POSITIONING, CONTROL	3–1/2 HOURS/RUN (3–6 RUNS REQ'D)	SPECIMEN PREP, TEST SET UP, ACTIVATE TEST EQUIPMENT, INITIATE TEST, RECORD TEST DATA, TERMINATE TEST,	GROUND LAB VACUUM CHAMBER 10^{5} TO 10^{-6} N/M^2	SPECIMEN PREP, TEST SET UP, ACTIVATE POSITIONING APPRATUS, MONITOR, CONTROL, TERMINATE TEST.
③ HEATING METHOD, EFFECTS OF INITIAL TUNGSTEN PURITY	4–5 HOURS RUN (6–12 RUNS REQ'D)	SPECIMEN PREP, TEST SET UP, INITIATE HEATING, RECORD PROCESS DATA, MELT, COOL. TERMINATE,	GROUND LAB VACUUM 10^{-3} TO 10^{-6} N/M^2 X–RAY SHIELDING	PRE– AND POST–TEST ANALYSES, SPECIMEN PREP, SET UP TEST, INITIATE HEATING, MONITOR AND CONTROL, MEASURE GAS DATA, INITIATE COOLING, COLLECT SPECIMEN.
④ HEATING, DEGASSING METHODS AND EFFECTS OF INITIAL PURITY	4–1/2 HOURS/RUN (6 RUNS REQ'D)	PRE–TEST MATERIAL CHARACTERIZATION, SPECIMEN PREP, TEST EQUIPMENT AND INSTRUMENTATION SET UP; INITIATE LEVITATION, HEATING; MEASURE GAS OUTPUT; CONTROL HEATING, DWELL, COOLING PROFILE; MATERIAL CHARACTERIZATION,	AS IN ③ PLUS LEVITATED SPECIMEN.	AS IN ③
⑤ PROCESSING STEPS AND CONDITIONS, DATA ACQUISITION AND EVALUATION EVALUATION	1 1/2–5 HOURS/RUN PLUS OPERATIONAL TESTING	AS IN ④ PLUS MELT AND SUPERHEAT, SUPERCOOL BEFORE COOLING, ALSO ADD OPERATIONAL TESTING.	AS IN ④	AS IN ④ PLUS FABRICATION OF PRODUCT INTO TARGET MOUNTED IN TUBE, INSTALL IN OPERATIONAL TEST RIG, INITIATE OPERATIONAL TEST, TERMINATE.

Figure III-82A. Summary Mission Profiles for Experiments in Support Of High Purity Tungsten X-Ray Targets

EXPERIMENT/TEST	DURATION	SEQUENCE OF FUNCTIONS	EXPERIMENT TEST CONDITIONS	CREW TASKS
DROP TOWER, KC–135, SOUNDING ROCKET EXPERIMENTS AND TESTS				
⑥ POSITIONING SYSTEM EVALUATION AND DESIGN DATA ACQUISITION	DROP TOWER, 3–10 SEC (10–20 RUNS REQ'D) KC–135, 20–40 SEC (10–20 RUNS REQ'D) SOUNDING ROCKET 4–10 MIN (1 RUN REQ'D)	POSITIONING TEST PACKAGE PREP AND INSTALLATION, CHECKOUT. INITIATE MISSION. INITIATE TEST (FOR KC–135, CONTROL PACKAGE POSITION). RECORD POSITION, VELOCITY, ACCELERATION DATA, TERMINATE TEST. RECOVER TEST PACKAGE.	~ ZERO "G" TEST PACKAGE, 10^{5} TO 10^{-3} N/M^2 ATMOSPHERE	TEST PACKAGE PREP, INSTALL, CHECKOUT, (IN KC–135, CONTROL PACKAGE). RECOVER.
⑦ PRELIMINARY VERIFICATION OF HEATING, DEGASSING METHOD	7–10 MIN/RUN (2 RUNS REQ'D)	SPECIMEN PREP, HEATING, DEGASSING TEST PACKAGE PREP AS IN ⑥ FOR SOUNDING ROCKET, PLUS HEATING DATA, GAS MEASUREMENTS.	~ ZERO "G" HEATING IN TEST PACKAGE TO ~ 2000 TO 2700°K	AS IN ⑥
⑧ PRELIMINARY VERIFICATION OF PROCESS	8–10 MIN/RUN	AS IN ⑦ FOR PROCESS PACKAGE.	~ ZERO "G" HEATING TO ~ 3700°K	AS IN ⑦
SHUTTLE SORTIE LAB TESTS				
⑨ PROCESS DESIGN VERIFICATION	1 1/2–3 HOURS/RUN PLUS 15 DAYS OPERATIONAL TESTING (3–6 RUNS REQ'D)	SPECIMEN PREP, PRE–LAUNCH, LAUNCH OPERATIONS, SPECIMEN INSPECTION. MEASURE PROCESS PARAMETERS. INITIATE POSITIONING, HEATING, ATMOSPHERE CONTROL, DEGAS, MELT, SUPERHEAT, SUPERCOOL, COOL. STORE PRODUCT. RECYCLE.	~ ZERO "G" HEATING ~ 3700°K X–RAY PROTECTION	PRE–TEST CHECKOUT, SPECIMEN INSPECTION AND REMOVAL. PROCESS INITIATION, MONITORING, CONTROL. SPECIMEN STORAGE. RECYCLE. TERMINATE. OPERATIONAL TESTING IN GROUND LAB.
⑩ PROTOTYPE TEST	AS IN ⑨	AS IN ⑨	AS IN ⑨	PRE–TEST CHECKOUT, MONITOR, MAINTAIN, REPAIR, TERMINATE. OPERATIONAL TESTING IN GROUND LAB.

Figure III-82B. Summary Mission Profiles for Experiments in Support Of High Purity Tungsten X-Ray Targets

initiated. Vacuum degassing data and experimental electromagnetic positioning of solid tungsten should be accomplished facts in the next several months.

The test conditions shown in the figure reveal one of the operational problems associated with certain of the required tests - the high temperatures associated with processing tungsten. Integrating such experiment and test operations into flight operations of the air-and space-borne facilities will require particular care and good design.

Note should be taken of the "Operational Testing" included in functions and crew tasks aspects of several tests. This testing, described in Figure III-69, is a ground laboratory test in common industry use for ascertaining life/performance of x-ray target materials produced by any method, not only those produced by the approach under study here.

III.9.4 MISSION PROFILES FOR EXPERIMENTS AND TESTS IN SUPPORT OF FABRICATION OF SURFACE ACOUSTIC WAVE COMPONENTS

While this study has not penetrated deeply into the processes for growing the required piezo and non-piezo crystals, our earlier discussions in Section III.4 have emphasized the requirements for extreme perfection, and have defined the specific materials of interest. We have also emphasized that such crystals should be obtained from whatever source available - ground or orbiting facility. Thus the mission profile chart, Figure III-83, reflects the timing, functions, conditions and tasks of both ground-based and space-based crystal growth. More significant, however, are the requirements for crystal characterization shown in the profiles of the ground testing. Such characterization will be performed on all potentially useful crystals.

It must be noted that testing on three process steps listed for short duration Zero "G" testing (crystal ultra-cleaning, metallization and resist-coating),

EXPERIMENT/TEST	DURATION	SEQUENCE OF FUNCTIONS	EXPERIMENT/TEST CONDITIONS	CREW TASKS
GROUND LAB EXPERIMENTS/TESTS				
① CRYSTAL GROWTH METHOD EVALUATION	2 DAYS/RUN (8–16 RUNS REQ'D)	PRETEST SPECIMEN ANALYSIS, CRYSTAL GROWING APPARATUS SET UP, INITIATE CRYSTAL GROWTH. MONITOR PROCESS PARAMETERS. MEASURE GROWTH RATE, COOL, X-RAY CRYSTALLOGRAPHY, MATERIAL CHARACTERIZATION.	GROUND LAB, HEATERS TO ~ 2500°K, CONTROLLED CRYSTAL GROWTH APPARATUS (CZOCHRALSKI, FLUX, EPITAXY, ETC.), INERT GAS 10^{-3} TO 10^{5} N/M^2 ATMOSPHERE.	PRE- AND POST-TEST SAMPLE ANALYSIS, SET UP TEST, INITIATE PROCESS, MONITOR AND CONTROL PROCESS, MEASURE GROWTH RATE, TERMINATE, CRYSTALLOGRAPHY.
② MEASURE VIBRATION IN MASK FABRICATION FACILITY	1 DAY RUN (4–8 RUNS REQ'D)	EMPLACE VIBRATION MEASUREMENT INSTRUMENTATION, INITIATE MEASUREMENTS, RECORD DATA, TERMINATE TEST.	GROUND FABRICATION FACILITY	INSTRUMENT EMPLACEMENT, INITIATE MEASUREMENTS, TERMINATE.
③ ASSESSMENT OF ELECTRON BEAM SIZE, VIBRATION, VIBRATION ISOLATION EFFECTS ON MASK FABRICATION EQUIPMENT	1 DAY/RUN (5–10 RUNS REQ'D)	SET UP ELECTRON BEAM GUN, SET UP VIBRATION ISOLATORS, INITIATE TEST CONDITIONS AND INSTRUMENTATION, INITIATE MASK FABRICATION, MONITOR AND CONTROL, TERMINATE TEST.	SELECTED GROUND FABRICATION FACILITY, FABRICATION CHAMBER WITH VACUUM SYSTEM, VARIABLE VIBRATION ISOLATION.	SET UP OF EQUIPMENT, ISOLATORS, INSTRUMENTATION; INITIATE TEST CONDITIONS, INSTRUMENTATION, TEST, MONITOR AND CONTROL TEST. TERMINATE TEST.
④ ASSESSMENT OF VIBRATION, VIBRATION ISOLATION EFFECTS ON SOFT X-RAY LITHOGRAPHY	~ MINUTE RUN (5–10 RUNS REQ'D)	ALIGN MASK, RESIST-COATED SUBSTRATE, INITIATE INSTRUMENTATION, EXPOSE, ANALYSIS AND MEASUREMENT OF PATTERN.	CLEAN ROOM, SHIELDING, X-RAY LITHOGRAPHIC EQUIPMENT, VARIABLE VIBRATION ISOLATION.	SET UP OF EQUIPMENT, ISOLATORS, INSTRUMENTATION, ALIGN MASK, SUBSTRATE, CONTROL VIBRATION ISOLATION, INITIATE EXPOSURE, ANALYZE AND MEASURE PRODUCTS.
⑤ PROCESS EQUIP. DEV. TESTING	1 MIN TO 5 DAYS (8–10 RUNS REQ'D)	FUNCTIONS VARY WITH EQUIPMENT UNDER TEST AND SPECIFIC TEST OBJ.	ENG'G LAB WITH TEST LOAD CONDITIONS.	SET UP, INITIATE INSTRUMENTATION, INITIATE TEST, MONITOR, CONTROL, RECORD DATA, TERMINATE.
DROP TOWER, KC-135 AND SOUNDING ROCKET EXPERIMENTS AND TEST				
⑥* CRYSTAL ULTRACLEANING METHODS AND PERFORMANCE	KC-135, 20-40 SEC/RUN (10–20 RUN REQ'D) SOUNDING ROCKET, 5–10 MIN/RUN (2 RUNS REQ'D)	LOAD ION-BEAM SCRUBBING TEST PACKAGE, INSTALL, INITIATE MISSION (IN KC-135, CONTROL TEST PACKAGE), INITIATE TEST, MEASURE PROCESS PARAMETERS, TERMINATE TEST, RECOVER PACKAGE, REMOVE AND ANALYZE CRYSTAL SURFACE, REPEAT FOR BACK SPUTTERING TEST.	~ ZERO "G," TEST PACKAGE VACUUM 10^{-3} TO 10^{-6} N/M^2.	INTEGRATE TEST PACKAGE, PRE-MISSION PREP AND CHECKOUT (IN KC-135, CONTROL PACKAGE), RECOVER PACKAGE AND TEST SPECIMENS.

**IF DECISION IS FOR PREFERRED IN-SPACE PROCESS STEP.

Figure III-83A. Summary Mission Profiles for Experiments in Support Of Fabrication of Surface Acoustic Wave Components

EXPERIMENT TEST	DURATION	SEQUENCE OF FUNCTIONS	EXPERIMENT TEST CONDITIONS	CREW TASKS
(7)** CRYSTAL SURFACE METALLIZATION METHODS AND PERFORMANCE	KC-135, 20-40 SEC/RUN (4-60 RUNS REQ'D) SOUNDING ROCKET, 1-10 MIN/RUN (2 RUNS REQ'D)	AS IN (5) FOR SPUTTERING TEST PACKAGE, AND FOR VAPOR DEPOSITION PACKAGE.	AS IN (6)	AS IN (6)
(8)** RESIST-COATING METHODS AND PERFORMANCE	DROP TOWER, 4-10 SEC/RUN (2-10 RUNS REQ'D) KC-135, ~20 SEC RUN (2-10 RUNS REQ'D)	AS IN (5) FOR "SPINNER" AND MODIFIED "SPINNER" TEST PACKAGES, VARIOUS COATINGS.	AS IN (6)	AS IN (6)
AUTOMATED SPACECRAFT EXPERIMENTS AND TESTS				
(4A) ASSESSMENT OF VIBRATION, VIBRATION ISOLATION	1 DAY/RUN (5-10 RUNS ON DIFFERENT SPACECRAFT REQ'D)	INSTALL VIBRATION-MEASURING INSTRUMENTATION ON SPACECRAFT, PRE-LAUNCH, LAUNCH OPERATIONS, INITIATE MEASUREMENTS ON-ORBIT, TELEMETRY OF DATA. REPEAT WITH VARIOUS MASSES MOUNTED ON VIBRATION ISOLATORS.	ORBITING ENVIRONMENT, VARIOUS SPACECRAFT REPRESENTING VARIOUS VIBRATION ENVIRONMENTS.	INSTALLATION, PRE-LAUNCH, LAUNCH OPERATIONS. TELEMETRY RECEPTION. ANALYSIS.
SHUTTLE-BASED EXPERIMENTS AND TESTS				
(1A) CRYSTAL GROWTH METHOD VERIFICATION	2 DAYS/RUN (4-8 RUNS REQ'D)	PRELAUNCH, LAUNCH OPERATIONS, CRYSTAL GROWTH EQUIPMENT START-UP, MONITOR, CONTROL CONDITIONS AND EQUIPMENT, RECORD DATA, MEASURE GROWTH RATE, COOL, TERMINATE TEST, RECOVER CRYSTAL, PACKAGE FOR RETURN.	TEMPERATURE, ATMOSPHERE CONSISTENT WITH SELECTED CRYSTAL MATERIAL AND GROWTH METHOD, ZERO "G".	PRELAUNCH, LAUNCH OPERATIONS, CHECKOUT, EQUIPMENT SET UP; CONTROL, MONITOR CONDITIONS, EQUIPMENT OPERATION. INITIATE TEST, MEASURE GROWTH, TERMINATE TEST, RECOVER AND PACKAGE CRYSTALS.
(9) PROTOTYPE DEMONSTRATION	2 DAYS/RUN (2-4 RUNS REQ'D)	PRELAUNCH, LAUNCH OPERATIONS, CHECKOUT, INITIATE PROCESS. PERFORM IN-SPACE STEPS, PACKAGE FOR GROUND STEPS.	AS IN (1A)	PRELAUNCH, LAUNCH OPERATIONS, CHECKOUT, INITIATE PROCESS, PERFORM MANUAL INTERFACE BETWEEN PROCESS STEPS. MAINTAIN, REPAIR, PACKAGE PRODUCTS.

**IF DECISION IS FOR PREFERRED IN-SPACE PROCESS STEP.

Figure III-83B. Summary Mission Profiles for Experiments in Support Of Fabrication of Surface Acoustic Wave Components

previously noted as potentially benefiting only slightly from space processing, are shown for planning purposes only. Final decisions will depend upon results of precursor ground tests and process implementation analyses.

III.9.5 MISSION PROFILES FOR EXPERIMENT AND TEST FACILITIES

The previously discussed experiment and test procedures (given in Figures III-32 to 47, III-49 to 57, III-59 to 69, and III-71 to 78), and the experiment and test mission profiles in Sections III.9.1 to III.9.4, must ultimately be coupled to the profiles of the facilities in which they will be performed.

The following paragraphs provide a review of the facility profiles, extracted from available documentation. Since this document cannot provide the volume for all pertinent data of all feasible facilities, we have chosen representative facilities and information, and list several sources of additional data.

Ground Based Laboratories

In the main, ground based laboratories contain such a variety of equipments and specialists that to provide a mission profile of all functions, operating conditions, and personnel tasks would comprise a report in itself. We have, therefore, elected to avoid providing total laboratory profiles, and instead, provide here significant profile data on a few laboratory facilities typical of aerospace industries, but likely to be less known in the industries involved in the products under study here.

a. Solar-Thermal-Vacuum Chamber (Ref. GE Document PIB-A-64)

   Test Package, up to 6.3M Diam. by 6M and 20,000 KG

   Vacuum , 10^{-4}, to 10^{-7} N/M^2

   Wall emissivity, .9 to .95

   Solar simulation, .2 to 2.5 microns, 15% uniformity

   Start-up-to-operating time, 7 hours to 10^{-5} N/M^2

   Shutdown and Warmup , 20-24 hours

   Instrumentation , 800 channels for thermocouples

b. Vibration Exciters (Ref. GE Document PIB-A-64)

Force Output , 55 to 1600 KG peak sine or random (RMS)

Stroke, 2.54CM double amplitude

Isolation, 568,000 KG seismic mass to 2.5 Hz

air mounted or seismic mass to 2 Hz

Instrumentation, 100 channel analog data

wide-band FM magnetic tape storage

10 channel real time data reduction

c. Large Centrifuge (Ref. Naval Air Development Center, Johnsville, Penna.)

Acceleration, up to 30 G (Programmable)

Test Package, 2M x 1M x 1M

Support to Test Package (during test), power, light, heat, communications

Instrumentation, 100 channel analog

Other potentially useful ground test facilities include neutral buoyancy tanks, anechoic chambers, and air bearing platforms.

Drop Towers

NASA operates two drop towers,

- The 89.6 Meter Tower at MSFC, and
- The Zero Gravity Research Facility at Lewis Research Center.

Key mission profile information on these facilities is given below:

a. Lewis Research Center Zero Gravity Research Facility (Ref. Lewis Research Center Brochure, "Zero Gravity Research" facility)

Duration of "Zero G" (1×10^{-5} G) - 10 sec. (up and down)

Experiment Package - 227 to 2720 KG

Up to 1.5M Diameter, 6M long

Initial Acceleration	- 40G
Average Deceleration	- 30G
Vacuum Pump Down Time	- $\sim$ 30 minutes
Test Support	- Clean Room (Class 10,000), (Class 100, on request).

b. MSFC 89.6 Meter Drop Tower (Ref. "A Review of Manufacturing in Space and Other Zero G Experiments being conducted in MSFC's 89.6 Meter Drop Tower" by V. H. Yost)

Duration of "Zero G" (1×10^{-5}G)	- 4.1 Sec. (Down Only)
Experiment Package	- Up to 204 KG
	- .91M x .91M x .45 to .91M
Maximum Deceleration	- 25G
Test Support	- Power, Telemetry (6 channels)

The following abstract from the referenced document details the major steps involved in a typical test sequence in the MSFC Tower.

1. The door of the drag shield is opened, the package is placed on the drag shield floor and the flat conductor cables that transmit power and sequencing signals are connected to the package and experiment.

2. The motor-generator set is started, and the package and experiment are sequenced to verify that they will operate properly during the drop.

3. The instrumentation system, which provides up to six channels for telemetering data from the experiment to the Saturn V West Area Block House, Building 4674, is tested to verify that it is operating properly.

4. The door to the drag shield is bolted closed and all personnel at the top of the Test Stand go into the operations room which is covered with steel.

5. The high pressure spheres in the top of the drag shield are filled with air to a pressure of approximately 1.21×10^7 N/m^2 (1750 psig), the thruster solenoid valve is opened and some air is discharged through the thrusters on top of the drag shield to verify that the system is operating properly, and the spheres are refilled with air to 1.21×10^7 N/m^2.

6. The air thruster solenoid valve is opened, the drag shield is released, and the thrust accelerates the drag shield down the guide rails faster than free-fall (9.8 m/sec^2 (32.16 ft/sec^2) . The result is that the drag shield floor is separated from the package and thepackage approaches free-fall (1×10^{-5} g) inside the drag shield for up to 4.135 sec or 89.6 m (294 ft) of the guide rails at which time the deceleration produced by the air drag and the friction between the guide rails and the drag shield is greater than the acceleration produced by the thrust and the package comes to rest on the drag shield floor.

7. The drag shield enters the top of the catch tube which compresses the air between the nose of the drag shield and the bottom of the catch tube. The compressed air is permitted to escape around the drag shield, which fits loosely in the catch tube, and through holes in the side of the catch tube at such a rate as to decelerate the drag shield (maximum deceleration is 25 g's) in 12.19 m (40 ft) to a low velocity before it comes to rest on a cushion of rubberized horse hair.

8. The cable is lowered with the winch to the top of the drag shield in the catch tube and attached to the release system on the drag shield. The drag shield is then pulled to the top of the guide rails where it is secured.

KC-135 Aircraft Flying Zero "G" Parabolas

While it is presently understood that this aircraft program is soon to be terminated, it was considered a possible candidate facility, based on the fact that well-experienced test operators with sufficient intestinal fortitude had acquired useful data in past programs.

Figure III-84 pictures the profile of a Zero "G" mission of the KC-135 aircraft, and key data is given below:

Available Test Volume in Aircraft - Cylinder 78" in diameter by 60 ft. long.

Duration of "Zero G" - 20 to 35 sec. (30 sec. average)

Available Electric Power - 10 watts @ 115v + Hz. Additional power may be available at 28v D.C. and at 400 Hz.

Cooling Available - By Special Arrangement

Maximum G Loading (Immediately preceding and following "Zero g"/ - 2.5 "G"

Test Operator Requirements - Must pass Class II FAA Flight Physical and Chest X-ray. Also must pass Survival School Training.

Lead Time (Between initiating plan and flight) - Variable, average 4-6 weeks.

Cost to Experimenter - $3,000 to $5,000 per Flight Hour.

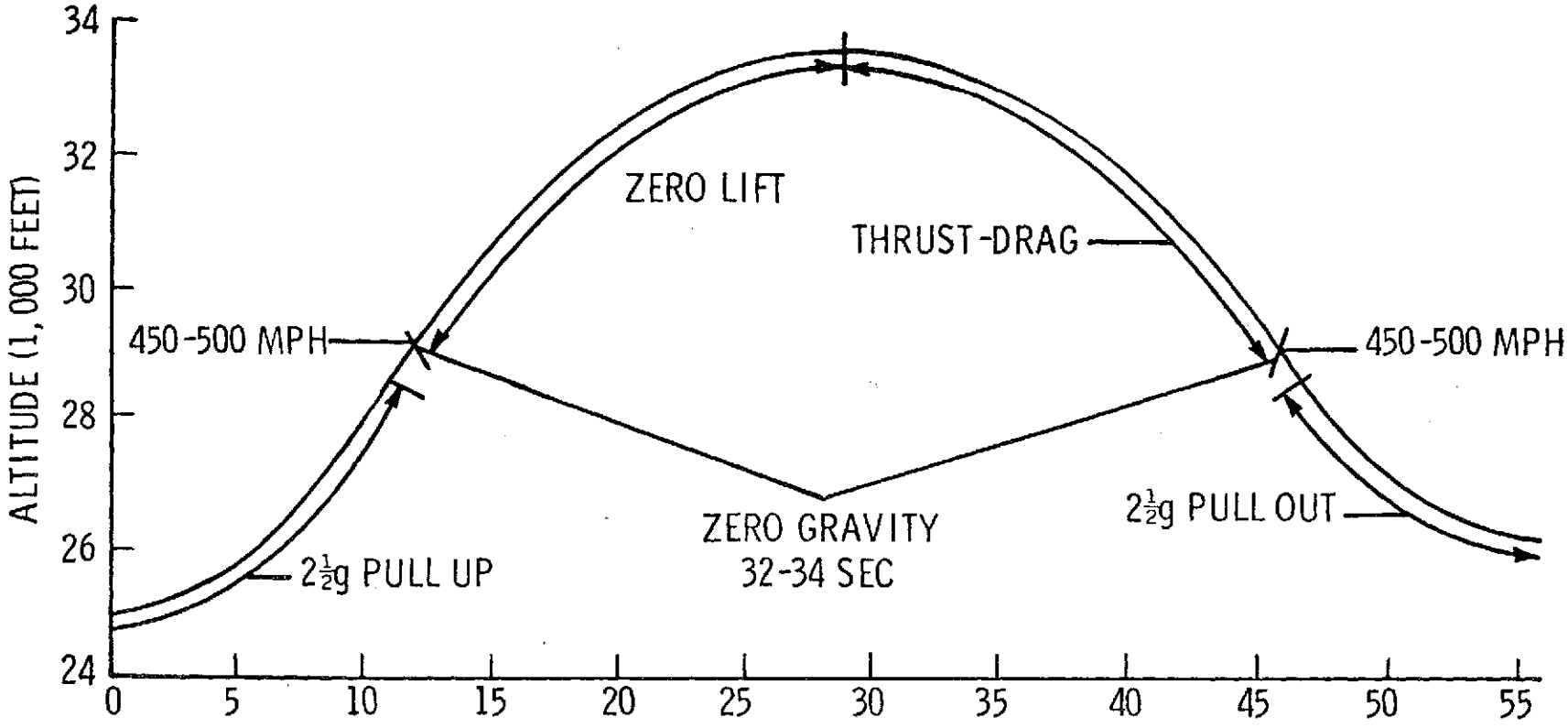
Number of Tests - 20 to 40 parabolas per flight,
Average of 5 flights per week.

Limitations on Experiments - Standard Safety Limitations (No flammables, Explosives, etc.)

Lead Time (Between arrival of experiment equipment and take off) - No Limits, Operation is simple bolt-down, strap-in.

Sounding Rockets (Many references. e.g. "The United States Sounding Rocket Program", Goddard Space Flight Center, X-740-71-337; Wendell H. Lee, Characteristics of Selected Launch Vehicles..., NASA Wallops Island Memo, Nov. 1973; L. Early, NASA Wallops; B.Montgomery, NASA MSFC, etc.)

The utilization of sounding rocket flights to provide modest durations of zero "G" for space processing is a relatively recent development (less than 3 years), but it has awakened sufficient interest to warrant major consideration here. Such an



MANEUVER OF KC-135 JET TANKER FOR PRODUCTION OF ZERO GRAVITY CONDITIONS.

Figure III-84. KC-135 "Zero G" Flight Profile

approach offers the desirable opportunity for low cost testing of selected Space Processing concepts during the interim period between Skylab and Shuttle.

Figure III-85 pictures the flight profile for a typical sounding rocket with indication of the effective Zero "G" duration for testing.

Other key mission profile data appear below:

- o System Elements - Booster, Sustainer, Payload Housing, Payload Support, Despin, Residual Motion Control, Recovery System.
- o Standard Operations - Launch Site (Typical, White Sands Proving Ground) Crew (Pre-launch, Launch, Recovery support)
- o Standard Payload Support - Telemetry, Power, etc.
- o Refurbishment, Reuse - Booster, (Sustainer), Payload Housing, etc.
- o Constraints

Acceleration, Deceleration Loads	- 10 to 30G
Vibration	- Up to 15G, 5-2000 Hz
Payload Weight, Size	- 20 to 300Kg, .063 to .38 m^3
"Zero G" (10^{-3} to 10^{-4}) Duration	- 0 to 9 minutes (except Javelin and Scout B)

- o Key Problem - "Coning" during "Zero G" flight. Simple despin may be insufficient to achieve better than 10^{-3}G. May require 3-axis attitude control, or new de-spin package.

Automated Spacecraft

At present, no automated spacecraft for Space Processing experiments and tests are planned prior to the Shuttle Operational date.

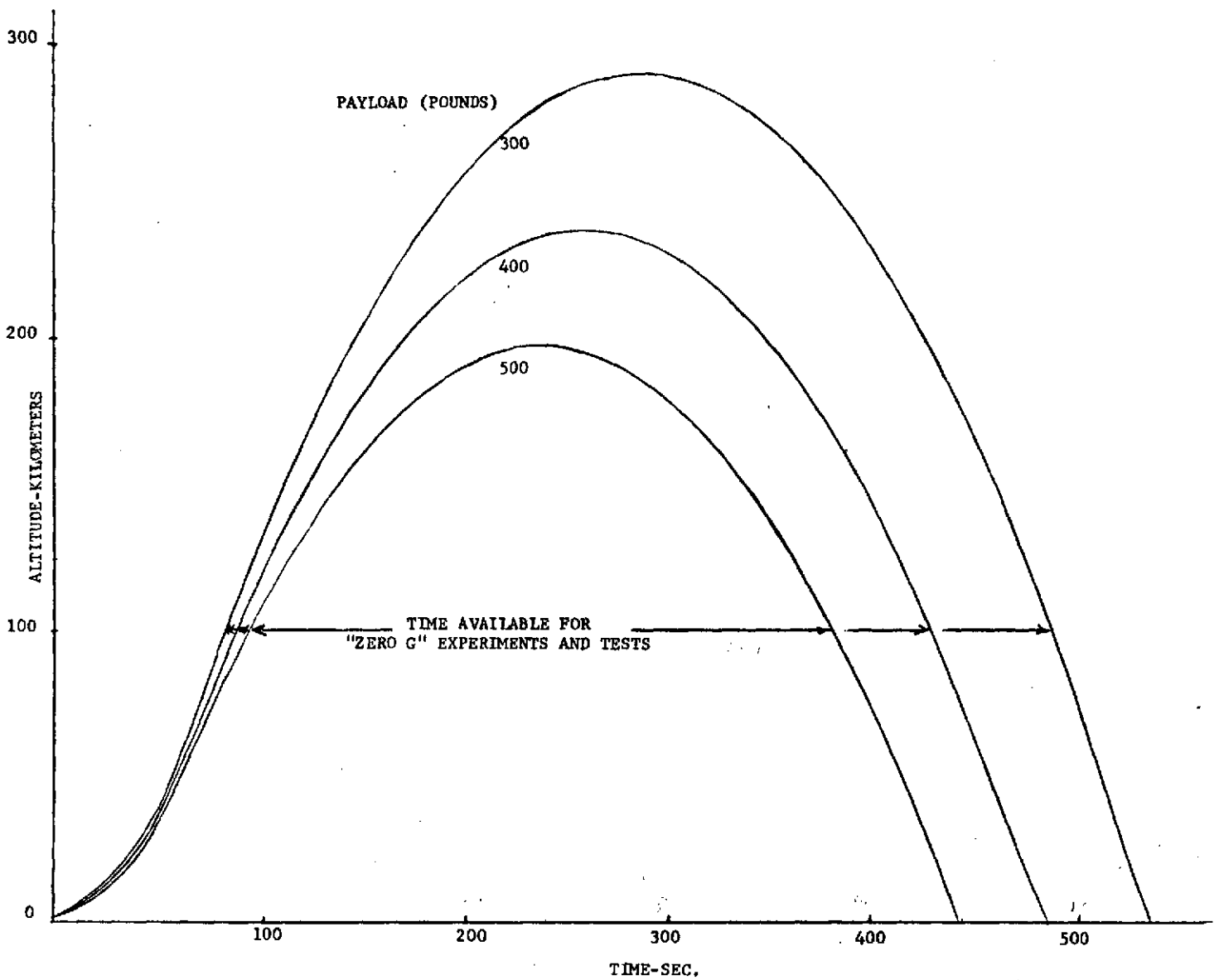


Figure III-85. Typical Aerobee 200 Trajectories

On the other hand, spacecraft for other missions are planned. While NASA-mission flights such as ERTS provide no capability for physical recovery of potential payloads, piggyback flights of non-recoverable payloads are in the realm of possibility. Such payloads would, of course, be constrained to non-interference with the primary mission, and be limited to telemetry of results.

Without specific spacecraft to consider, the general data for mission profiles are simply unlimited Zero "G" time, and, because of "piggyback" limitations, small experiment and test weights, volumes, power and communications.

Once the Shuttle initiates operations, however, automated spacecraft for space processing, if required, can be structured to a wide variety of mission profiles, limited only by those constraints and capabilities given for the Shuttle in the following paragraphs.

Space Shuttle

The Space Shuttle, with various combinations of Spacelab elements, is expected to be the workhorse of Space Processing (and other) missions. Not only will larger Space Processing Experiment and test payloads be possible, but investigators will have the opportunity (directly or through the crew) to adjust, modify, or develop experiments and tests, inflight.

A typical mission profile for the Space Shuttle, adapted from JSC 077000 "Space Shuttle System Payload Accommocations" is given in Figure III-86.

Figure III-87 is a copy of typical mission profile data presented in the above document, and is representative of the detailed information available there.

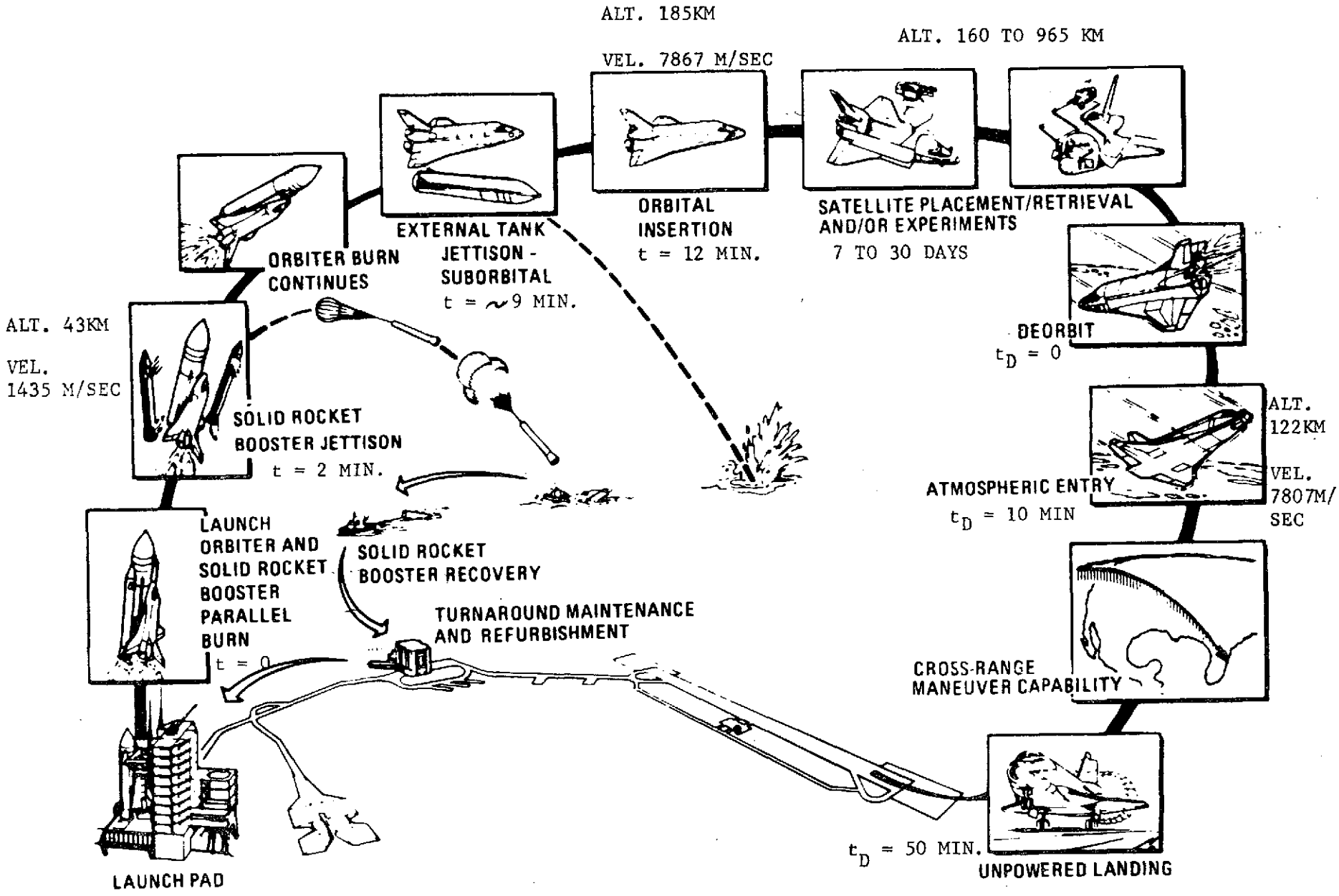


Figure III-86. Shuttle Mission Profile (Typical)

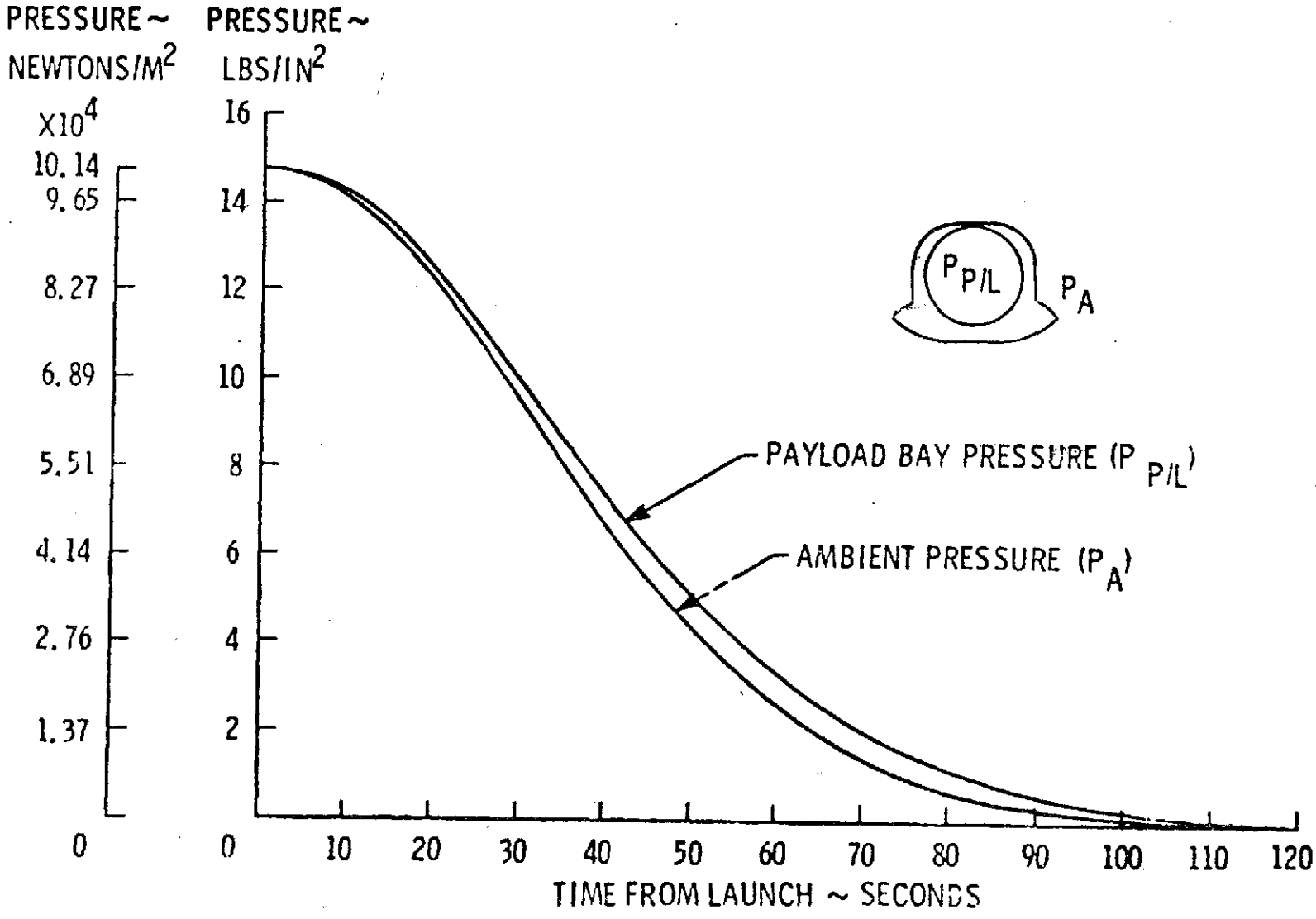


Figure III-87. Payload Bay Ascent Pressure History

III.10 TIMELINES AND MILESTONES OF PRE-PRODUCTION RESEARCH AND DEVELOPMENT PROGRAMS

The experiments and tests discussed in the previous sections are major technical considerations in the development of the four products under study. Such products, however, also require planning information prior to initiation of effort to develop them.

The keystone of planning a development program is a schedule of the major activities and events organized into the logical combination of parallel and sequential steps that culminates in the initiation of full-scale production. Such a schedule forms the "roadmap" for a development plan, which, as noted in Reference (8), provides the commercial User with necessary data to perform his future business planning. The relationship of the development plan to resources planning and marketing is given in Figure III-88.

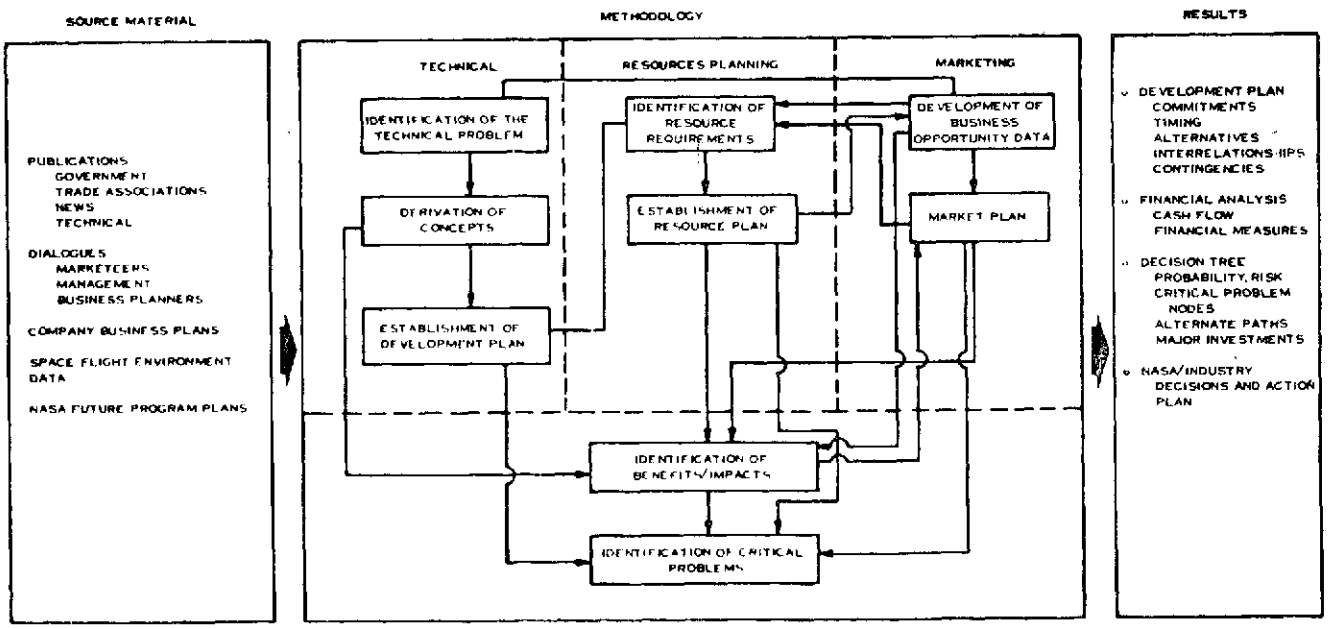


Figure III-88. Future Business Planning Methodology

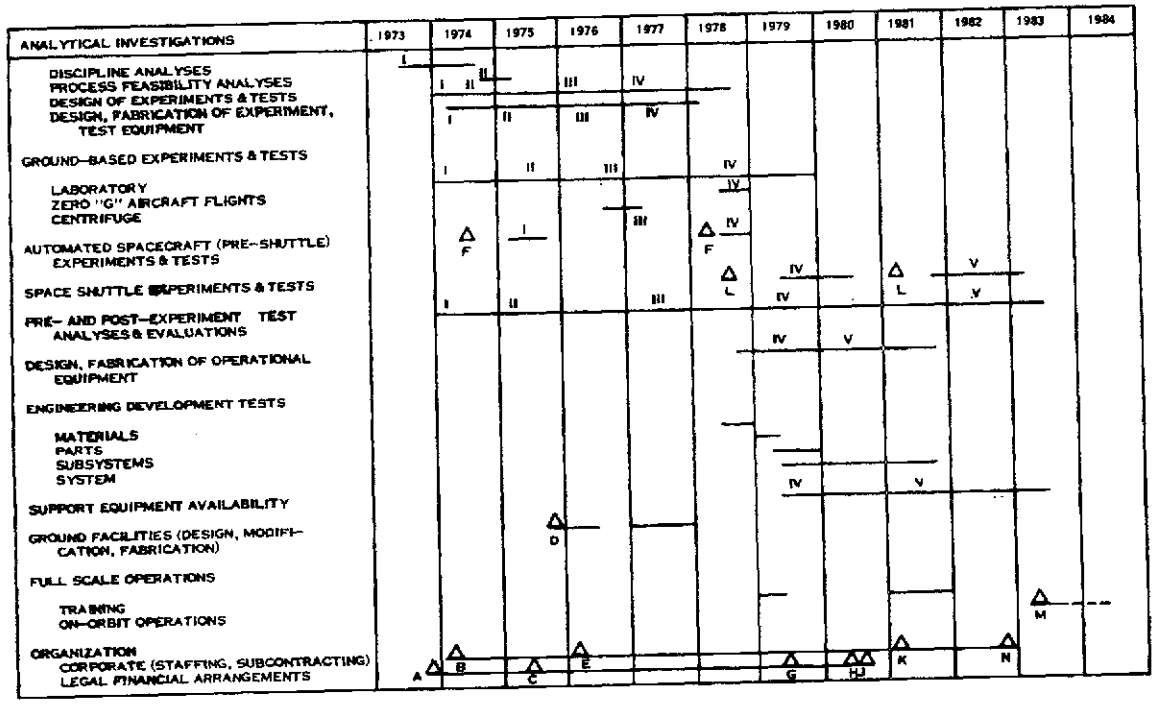
(8) Scarff, D. D. and Bloom, H. L.; "A Businessman Views Commercial Ventures In Space", AIAA Paper 73-75, January 1973.

III.10.1 TIMELINES AND MILESTONES FOR RESEARCH AND DEVELOPMENT TO ENABLE HIGH SPECIFICITY SEPARATION OF ISOENZYMES

Figure III-89 reveals that the User has placed a high emphasis on the acquisition of phenomenological data in the early stages of his program. The rationale for that view is that, if the full benefits of Zero "G" are to be realized for the electrophoretic/isoelectric focussing separation of isoenzymes, a number of changes in the present processing methods should be considered. For instance, present electrophoretic separation utilizes a voltage gradient of 10 volts per centimeter, a gradient which is larger than what is easily tolerable by some isoenzymes without denaturization. Smaller gradients, which lead to other problems of heating and convection in ground-based separations, should be feasible in space processing. What must be established in early analyses and experiments, is the mobility of isoenzymes under lower voltage gradients, and the selection of gradients for further development. Similar rationales apply to separation path length, gel pore size, and convection data.

The figure also demonstrates the progression of research on phenomenology to research on variations in the methods of separation (electrophoresis versus isoelectric focussing) to acquisition of development data for equipment (including evaluation of such available ground-based equipment as freezers, separators, to developmental testing of new or modified equipment and verification of its performance, to the final development step, prototype testing.

As indicated in the figure, the progressive steps leading to this final proof testing involve progressively more sophisticated facilities, with the Space Shuttle anticipated to provide key developmental data and proof testing in 1979 to 1980, and 1981 to 1983, respectively.



REMARKS

TEST SERIES

I – PROCESS PHENOMENOLOGY
MOBILITY EFFECTS
CONVECTION EFFECTS
PATH LENGTH EFFECTS

II – SEPARATION EVALUATION
SELECTION OF METHOD
DEMONSTRATION

III – NON-SPACE ENVIRONMENT EFFECTS
LAUNCH
STORAGE

IV – DESIGN DATA AND VERIFICATION
OPERATING ENVIRONMENTS EFFECTS
PERFORMANCE DATA

V – PROTOTYPE TEST
PROOF TEST

MILESTONES

A – FUNDING ARRANGEMENTS SETTLED – TEST SERIES I, II, III

B – STAFF ADDED FOR I, II, III

C – FUNDING ARRANGEMENTS SETTLED FOR TEST SERIES IV

D – LAB SPACE ADDED FOR IV

E – STAFF ADDED OR SUBCONTRACT FOR IV

F – SPACE RESERVED FOR EXPERIMENT OR TEST ON AUTOMATED SPACECRAFT

G – PATENT RIGHTS SETTLED FOR IV EQUIPMENT ON SHUTTLE

H – PRODUCT MARKETING ARRANGEMENTS SETTLED WITH NASA

J – FUNDING ARRANGEMENTS SETTLED FOR V

K – MAKE BUY DECISION ON INITIAL BIOLOGICAL

L – SPACE RESERVED ON SHUTTLE FOR TEST

M – START OF PRODUCTION

N – ARRANGEMENTS SETTLED FOR PROCESSING OF SPACE PRODUCT

Figure III-89. Timeline and Milestones for R&D Program For Separation of Isoenzymes

We have utilized the specific product know-how of the Users participating in this study, and our own space expertise to synthesize timelines and milestones, the "roadmap", for developing the four products under study.

Our Space Division Study Team members established groundrules, guidelines, capabilities and constraints of space operations and space programs, while, in a parallel effort, the Users defined ground laboratory, analytical, engineering, and organizational elements. Continuing our previously-developed relationships and direct dialogs with the Users, we then melded these outputs into a contiguous schedule by shifting time slots, establishing parallel efforts where feasible, combining experiments and tests, or parts thereof, where feasible, and restructuring activities where potential savings might accrue with no loss in technical content.

The resulting timelines and milestones appear technically satisfactory from both the space and commercial viewpoints. Required space program capabilities are within the realm of reasonable planning, and, aside from the fact that Users would like to see the development program start today, the schedules appear "comfortable". That is, sufficient time appears to be available to perform all listed activities, and sufficient durations between key test series are available to analyze experiment and test results and to restructure subsequent efforts. Typically, the experiment and test requirements discussed earlier have identified automated spacecraft facilities for some tests prior to Shuttle flights. While some such experiments and tests could possibly be accommodated as "piggyback" experiments on other primary missions, the possible unavailability of such accommodation, or of new spacecraft does not negate the planned development. The scheduling could accept early Shuttle flights or use of the Long Duration Exposure Facility as possible facilities for experiments and tests, with either no, or little delay in the overall product development.

The number, type, and importance of experiments and tests tend to fix attention on that aspect of R&D. The more prosaic problems, however, of basic materials and hardware acquisition, availability of support equipment, and establishment of ground facilities, form a significant portion of the R&D effort. Those elements are shown in the figure as requiring early attention. The User has indicated here, for instance, the need for additional laboratory space late in 1975 and in 1977.

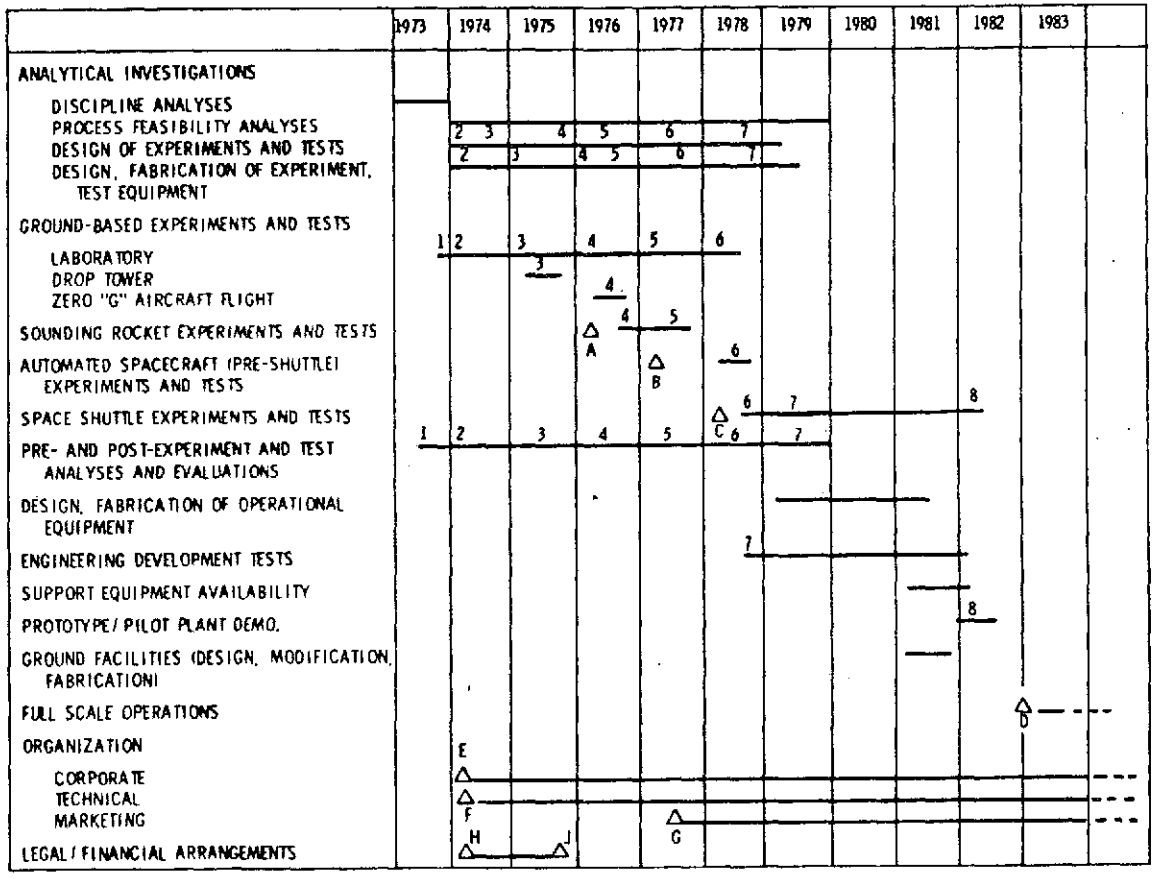
Provision is also made for the 1979 and 1981 training of space processing crews to carry out the testing on board the Shuttle in 1979 and 1980, and to commence full-scale operations in 1983.

An initial identification of non-technical activities and events is also shown in the figure. Such activities as staffing the Users' facilities for the listed activities, and arriving at patent and financial agreements with NASA are major milestones the User foresees as early as 1973, 1974.

III.10.2 TIMELINES AND MILESTONES FOR RESEARCH AND DEVELOPMENT TO ENABLE PROCESSING OF TRANSPARENT OXIDES

Figure III-90 presents our current view of the research and development program required to achieve production of Transparent Oxides of Aluminum, Zirconium and/or Yttrium. The short duration of discipline investigations is indicative of the Users' view that the existing background of analysis is essentially sufficient to warrant initiation of analyses and experiments aimed at defining process requirements.

Thus, after initial experiments to characterize the available qualities and forms of Yttria, Zirconia and Alumina, the next three series of experiments; carried out in Ground Laboratory, Drop Tower, Zero "G" Aircraft and Sounding Rockets, are



REMARKS

TEST SERIES

1. CANDIDATE OXIDES EVALUATION
   TRACE IMPURITIES ANALYSIS
   X-RAY CRYSTALLOGRAPHY
2. PROCESSING CONDITIONS
   DEGASSING, PHASE CHANGES
   PROCESS ATMOSPHERE
   ENVIRONMENT FORMULATION
3. HEATING AND MELTING METHODS
   TECHNIQUES
   RESPONSES
4. LEVITATION, POSITIONING METHODS
   TECHNIQUES
   SYSTEM PERFORMANCE
5. INITIAL PROCESS FEASIBILITY
   EVALUATION
   DEVELOPMENT
   DESIGN DATA
6. FORMING METHODS
   TECHNIQUES
   RESPONSES
7. DESIGN DATA AND VERIFICATION
8. PROTOTYPE TEST
   PROOF TEST

MILESTONES

A. SOUNDING ROCKET FLIGHT APPROVED
B. TEST ACCOMMODATION ON SPACECRAFT APPROVED
C. ACCOMMODATION ON SHUTTLE APPROVED
D. START OF PRODUCTION
E.) ADMINISTRATIVE, TECHNICAL TEAM
F.) FORMED TO INTERFACE WITH NASA
G. MARKETING ANALYSES INITIATED
H. LEGAL, FINANCIAL ARRANGEMENTS INITIATED
J. FUNDING, PROPRIETARY RIGHTS, PATENT RIGHTS SETTLED

Figure III-90. Timelines and Milestones for R&D Program For Transparent Oxide Processing

devoted to acquiring data on heating and positioning techniques, as well as the oxides responses to variations in the processing steps involved in the selected approach.

By early 1977, these efforts are expected to culminate in test versions of such equipment as the positioning system, and in Ground Laboratory and Sounding Rocket tests to assess the feasibility of, and evaluate the potential performance of, such equipment.

Subsequent laboratory and spacecraft testing of key developmental equipment lead to final design data acquisition and processing system verification testing aboard the Shuttle in late 1979, which allows final design, fabrication and proof-testing of the prototype processing system in the Shuttle in 1982.

The anticipation that the system for processing transparent oxides is likely to be automated, has led us to bypass the need for operator training, for the time being. Results of testing in the 1978-79 time period should confirm this approach, and, if necessary, such training could still be completed in time for prototype testing.

The User has identified early (1974-75) milestones in the non-technical organizational and legal/financial areas, and decisions related to these milestones will be discussed in Section III.11.

III.10.3 TIMELINES AND MILESTONES FOR RESEARCH AND DEVELOPMENT TO ENABLE FABRICATION OF HIGH PURITY TUNGSTEN X-RAY TARGETS

Figure III-91 indicates the earliest current activities of all the four products under study. In anticipation of a contract[9] from NASA-MSFC, preliminary

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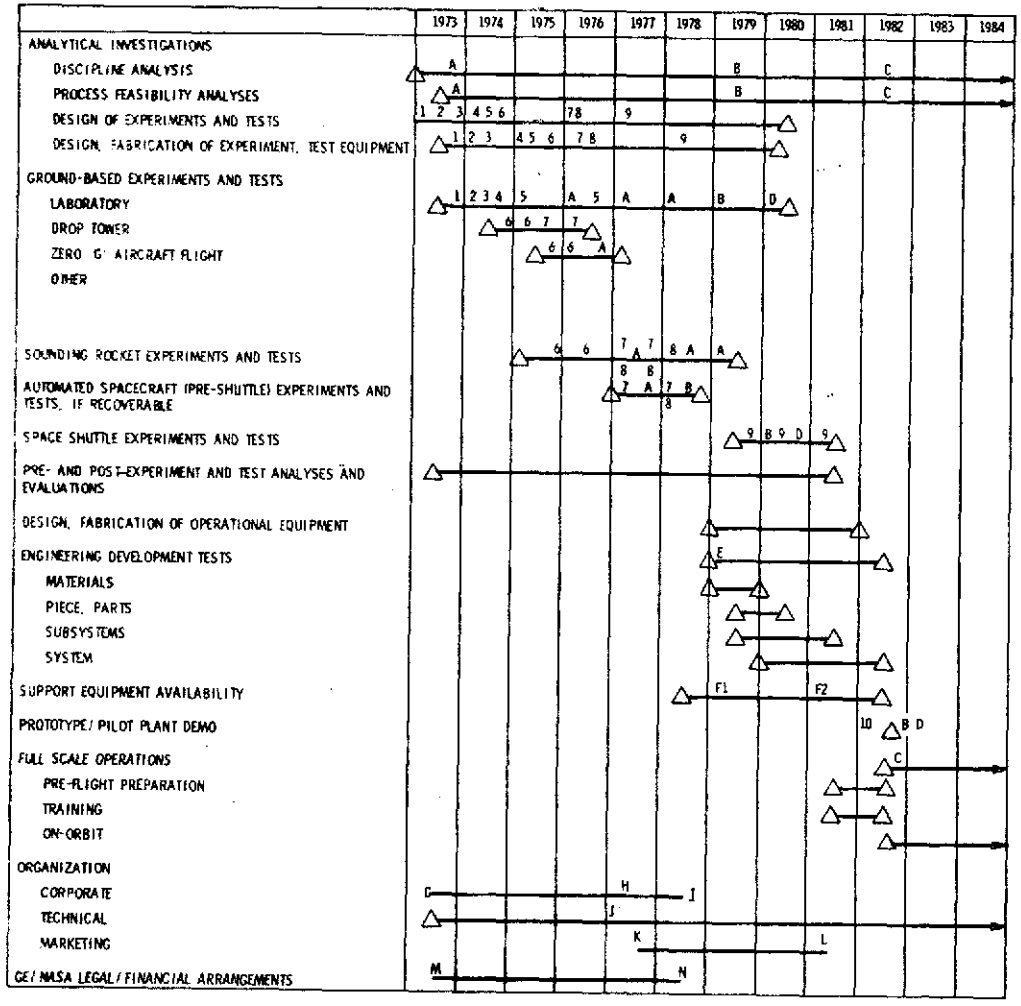
(9) "Development of Containerless Process For Preparation of Tungsten With Improved Service Characteristics"; General Electric Space Sciences Laboratory Proposal Number N25066, May 30, 1973.



REMARKS

TEST SERIES

1. LABORATORY VACUUM DEGASSING STUDIES
2. LABORATORY LEVITATION OF SOLID TUNGSTEN
3. LABORATORY ELECTRON BEAM HEATING STUDIES
4. LABORATORY VACUUM DEGASSING WITH LEVITATION
5. LABORATORY LEVITATION HEATING AND MELTING IN ONE G
6. DROP TOWER, ZERO "G" AIRCRAFT, SOUNDING ROCKET TESTING OF ZERO G POSITIONING DEVICE
7. DROP TOWER, SOUNDING ROCKET, SPACECRAFT TESTING OF VACUUM DEGASSING
8. SOUNDING ROCKET, SPACECRAFT TESTING OF DEGASSING, MELTING AND SUPERCOOLING
9. SHUTTLE EXPERIMENT TO FINALIZE PROCESS PARAMETERS
10. PROTOTYPE TEST

MILESTONES

A. PHENOMENOLOGICAL TESTS
B. UNIT OPERATION TESTS
C. PRODUCTION
D. UNIT OPERATION PROBLEMS TESTS
E. ENGINEERING DEVELOPMENT TESTS
F. SUPPORT AVAILABILITY
   F1 - PRIMARY EQUIPMENT
   F2 - SECONDARY EQUIPMENT
G. LIMITED FINANCIAL COMMITEMENT BY GE
H. BUSINESS PLAN ESTABLISHED
I. FUNDING, FINANCING DECISION
J. TECHNICAL FEASIBILITY DECISION
K. MARKET FORECAST
L. MARKET PLAN
M. INITIAL DEFINITION OF ISSUES ON PROPRIETARY RIGHTS, PATENTS, ETC.
N. SETTLEMENT OF PATENT AND FINANCIAL ARRANGEMENTS

Figure III-91. Timeline and Milestones For R&D Program For High Purity Tungsten Targets

discipline analysis and experiment design effort has been initiated, with the participation of the User, GE's Medical Systems Business Division.

The User anticipates a continuing analysis effort throughout the life of the R&D program as, from past experience, he recognizes new problems and approaches arising, which could contribute to improved tungsten properties, and/or larger size boules, and/or gains in shaping the product in zero "G".

Laboratory experiments are primarily aimed at acquiring basic information on fundamental processing steps; degassing, levitation, electron beam heating (as a possible supplement to electromagnetic heating), and combined processing effects.

Drop Tower tests could provide useful data as early as 1974, while Zero "G" aircraft and Sounding Rocket tests in 1975 and 1976 are programmed for key positioning system tests. Sounding Rocket tests form the backbone of testing the major process steps (positioning, degassing, melting and supercooling) in 1975 to 1978. The low cost aspect of such testing is very attractive to the User.

Automated spacecraft tests for tungsten processing development are valuable only if recovery is possible. If the indicated 1977, 1978 tests in such a facility are not possible, then they should be delayed to the 1979, 1980 Space Shuttle timeframe, with resultant delay of the Shuttle tests on final processing parameters. Such a delay should not affect the prototype proof-testing aboard the Shuttle in 1982, since design, fabrication, and testing of the prototype equipment is planned for a comfortable 3½ year schedule.

A current committment of GE-financed materials, test equipment and labor in support of early testing is typical of the non-technical activities shown in the figure. Other such activities and events, including proprietary rights and patent agreements are scheduled for negotiations and reviews in the 1973 to 1978 timeframe.

III.10.4 TIMELINES AND MILESTONES FOR RESEARCH AND DEVELOPMENT TO ENABLE FABRICATION OF SURFACE ACOUSTIC WAVE COMPONENTS

Since the User is basically attempting to extend the capabilities of already-existing disciplines and technology, no discipline analyses are anticipated, and only limited process feasibility analyses are listed, with most analytical work (other than design) being performed on experiment and test data.

The User admits, however, that should ground-grown crystals prove inadequate in perfection or size, and initial tests of space grown crystals (as in Skylab program) indicate significant advantages, then the analytical work on convective flows, such as that at the National Bureau of Standards[10] should likely be expanded.

The program represented in Figure III-92 includes a number of experiments and tests on process steps for which the space environment offers little or no benefit. The User has included them because the complete processing approach must intersperse such steps among those which do benefit from the space environment. Experience has shown that extensive handling of crystals, transportation after ultra-cleaning, metallization, etc. tends to result in an inordinate number of component failures. Thus, experiments and tests on cleaning methods,

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(10) "Investigation of Convective Effects and Crystal Growth", NBS, Contract Number W-13475

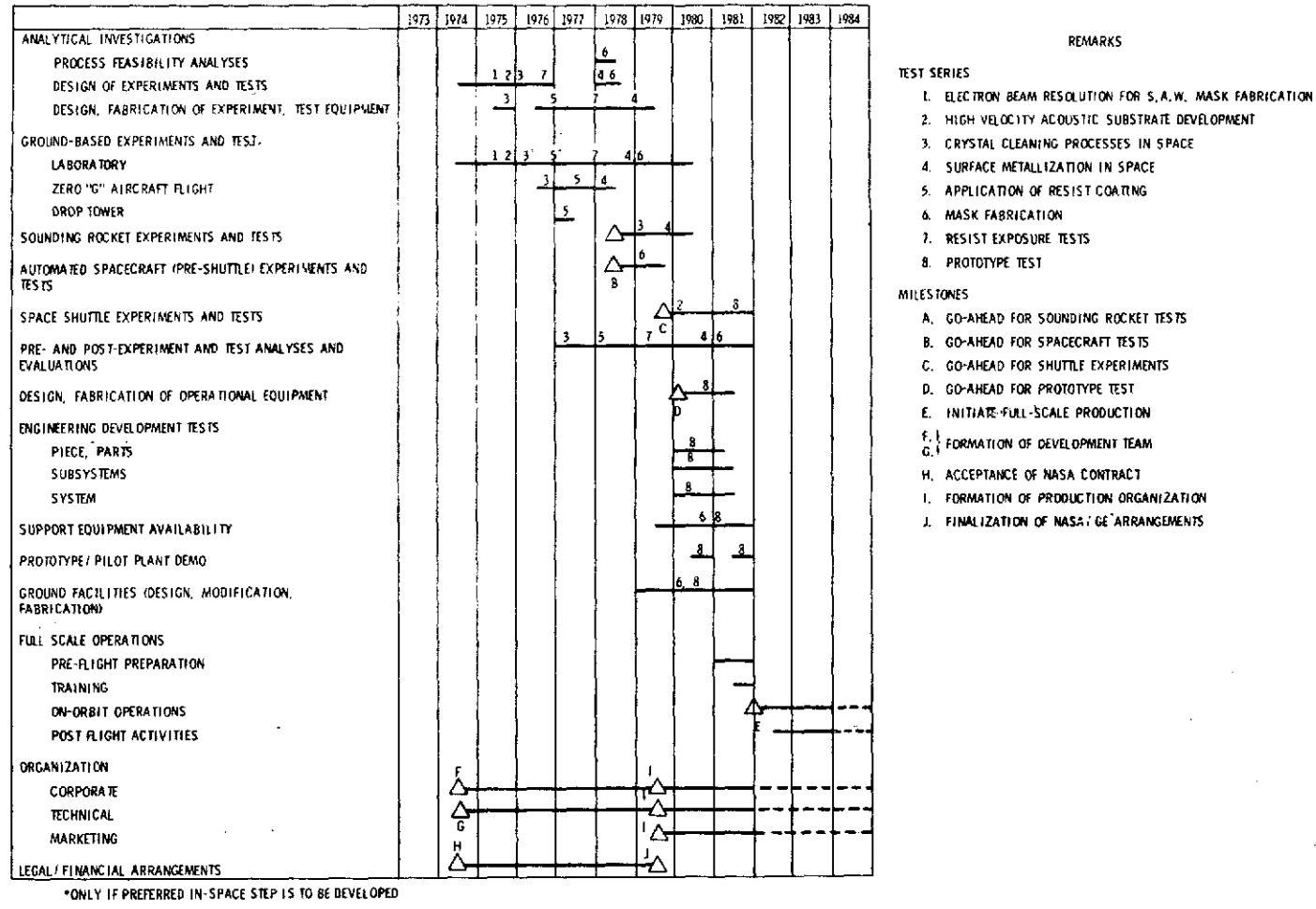


Figure III-92. Timeline and Milestones for R&D Program for Fabrication of Surface Acoustic Wave Components

metallization, and resist-application in Zero "G" aircraft, Drop Tower, and Sounding Rocket are listed provisionally, until such time as the decisions on ground-grown versus space grown crystals, economic impact, and process feasibility are made.

While not shown on the figure, the User has suggested a program related to this, and possibly other R&D efforts - the measurement of low frequency vibrations in current orbiting spacecraft. More discussion of this suggestion is found in Appendix D, Book 2, Volume II of this report. While not directly applicable to this program, such data would be useful in prognosticating the vibration levels needed in the feasibility analysis shown in 1978, and in establishing test parameters and vibration isolators for the Automated Spacecraft and Shuttle tests in 1979-1981.

The mask fabrication process step is the most critical in the selected process, and because of its duration (up to 40 hours), it requires long duration Zero "G" for verification testing - thus implying an Automated Spacecraft or Shuttle test. Should the planned Automated Spacecraft not be available, then the 1980 prototype test should be delayed to 1981, and the 1980 flight used for the mask fabrication test.

Much of the selected approach appears to be amenable to automation, thus the User feels little operator training is required.

The organizational and legal/financial activities should be addressed soon, but finalization of such activities is not warranted by the User to be sufficiently pressing to require action before 1979.

III.11 DECISIONS REQUIRED TO CARRY OUT SPACE PROCESSING PROGRAMS

The timelines and milestones appearing in the figures of Section III.10, in addition to identifying specific technical and management activities and their sequence, imply another major influence on the illustrated programs - decisions.

Some of the more straightforward decisions include: when to carry out a specific experiment or how many of a given test series shall be carried out. Less straightforward are decisions implied by "Were the results of Test A sufficiently good to warrant going on to Test B?", or "Shall we commit the dollars for this facility at this time?".

Furthermore, equally as important as the individual decisions is the role of any decision in the total decision flow. Some decisions are bound to be more critical than others. Furthermore, it is instructive, particularly for the commerically oriented products in this study, to document the available alternatives for such decisions.

In this final task of the Phase II Study, we undertook to identify the decisions that the Users felt were needed in carrying out the effort indicated by the previously documented R&D Program timelines and milestones. Our aim was to obtain the viewpoints of commercially oriented Users faced with a potentially profitable process, but also faced with becoming "involved" with two factors, new to his area of business - Space and the Government (NASA).

The study team has previously explored such relationships with various interested parties - the Users in both the Phase I and Phase II portions of this study, the GE executives comprising the Advisory Group for this study, and members of NASA's Advisory Board for this study.

From such dialogs have emerged a number of key points, many of which will be noted shortly in the decisions documented for the four product areas. Such points include proprietary rights, patents, funding, availability of facilities (mainly Shuttle).

It has also been pointed out that such issues hinge on establishing a viable Government/Industry relationship vis-a-vis commercial space processing.

A number of permutations and combinations of organizational entities could likely fulfill such a requirement, and Figure III-93 presents a matrix of alternative possible relationships among Government, Private Comany, and a Regulated Company patterned after COMSAT in which interested private companies could own stock. The various responsibilities and the ownership of products form one side of the matrix, while the other side reflects some opinions as to potential benefits and drawbacks of each arrangement, insofar as competitive commercial companies might view them.

The figure does not list all possible arrangements, but does, we believe, contain most of the reasonable combinations.

The first alternative, essentially the current mode of space processing activities, is that in which the Government (NASA) provides the transportation system, the processing facility, processing equipment (usually through funding contracts to companies), and the operations to carry out the mission. Since the Government provides and/or funds such activities and hardware, it is only to be expected that resulting products should be Government (thus publicly) owned. While such an approach requires little or no private investment, it will not be favored by private companies interested in commercial products because the essence of our free enterprise system is competition and profit. Commercial industries are in business to make money, and they make the most money when they can turn out a product which is theirs

ALTERNATIVE GOVERNMENT/INDUSTRY RELATIONSHIPS
IN COMMERCIAL SPACE PROCESSING

OPERATIONS EQUIPMENT ALTERNATIVE	PROVIDE TRANSPORTATION (SHUTTLE)	PROVIDE MODULAR FACILITY	PROVIDE PROCESSING EQUIPMENT	PROVIDE OPERATIONS	OWN PRODUCTS	PERFORM SPACE R&D	PERFORM GROUND R&D	REMARKS
I	G	G	G	G	G	G	G	Generally present mode, minimum co. investment. Not palatable to commercial ind.
II	C	C	C	C	C	C	C	Maximum protection of co. rights. Maximum co. investment. Can Co. utilize full Shuttle payload.
III	R	R	R	R	R	R	R	Less Co. investment. Less Co. payoff. Like ComSat. No private Co. proprietary rights. Can Reg. Corp. utilize complete Shuttle P.L.?
IV	G	R	R	R	R	R	R	Shuttle can also carry other P.L. Reg. Corp. pays proportionate transportation costs. Investment payoff, prop. rights and and competition as in III.
V	G	C	C	C	C	C	C	Shuttle can carry other P.L. Good protection of Co. rights. Co. pays proportionate transp. costs. Less investment. High payoff.
VI	G	G	C	G	C	G/C	C	With proper agreements, revised NASA rights clause: Min. invest., max. payoff, good Co. rights protection, open competition, Co. pays Govt share of transp. or royalties. Priority problems, Govt. "interference".

LEGEND: G, Government Responsibility, e.g., NASA
C, Private company responsibility, e.g., GE
R, Regulated Corp.; ala ComSat - GE, et al, public own stock

Figure III-93. Alternative Government/Industry Relationships In Commercial Space Processing

alone, or better than anyone else's. Typically, a six month's lead on the competition or a patent-protected product are aims for such industries. Government (public) ownership of space-produced products would inhibit such aims. Furthermore, Government investment for involvement in Alternative I would be high.

In the second alternative, we took the antithesis to the first alternative - a private company providing (and paying for) all aspects of their space processing program, including the transportation system. While the company's rights (thus competitive position) are well-protected in this alternative, the company's investment is highest, and it is questionable as to whether many companies can beneficially utilize the total Shuttle payload.

The third alternative postulates the formation of a regulated corporation, chartered and regulated by the Government, which would carry out all space processing activities and furnish resulting products to stock-owning companies for their business uses. While details of such a corporation require further thought, there could be some analogy to COMSAT or the TVA. While both Government and individual company investments might be minimized, some of the gross profit from space processed products would be absorbed by the regulated corporation, thus reducing private company profits. Most important to the private companies, the proprietary and patented techniques and equipment would necessarily be either developed by,or disclosed,to the regulated company (in order to carry out the actual processing). There is also the possibility that even the combined companies holding stock in the regulated corporation could not utilize full Shuttle payload capability.

Other alternatives are possible, and we have listed three in which the Government (NASA) owns and provides the Transportation System (Shuttle). Such an arrangement simplifies the mechanism by which Shuttle payloads may be fully utilized, that is, NASA simply adds various of their research modules in the Shuttle bay, so that the research modules and space processing manufacturing facility module add up to the Shuttle capability.

In these last three listed alternatives, the non-Government Users (the Regulated Corporation or the Private Company) would pay NASA a previously arranged portion of the operating costs. Such an approach provides an equitable means for defraying the Government's cost of space transportation without burdening a User with the total cost (unless the User utilizes the total payload capability) of operations.

The same remarks listed earlier for Regulated Corporation versus Private Company hold for these three alternatives. One additional remark is noted in the last-listed alternative. There is some thought required on the Company/Government relationship in performing the space-based research and development leading to commercial space processed products. While, at present, it is important for the Government (NASA) to lead the way in searching out new space processing disciplines, techniques, equipment, and materials, there is a point in the development cycle where commercial companies must begin to take on the responsibility (and funding) of such development. On the one hand, that transfer of responsibility is driven by economics - 1) NASA's budget is limited, and 2) it is only reasonable that companies which will be earning profits from products manufactured by space processing should fund required research. On the other hand, if NASA funds Research and Development, NASA should decide what should be done, and when. Companies may not always agree with such decisions. Some balance of Government and commercial company involvement (and funding) is therefore called for.

The preceding discussion will be reflected in various business-type decisions noted in the following sections, and relationships between business decisions and technical decisions will be illustrated in the accompanying decision flows. Particular note should be made of "Nodes" in the decision flows - points where decisions converge, where a single, or a few, decisions influence most of the subsequent decisions.

III.11.1 DECISION FLOW FOR SEPARATION OF ISOENZYMES

We have reviewed the timelines and milestones of Section III.10.1 to extract the decisions that will be required to carry out that program. With Polysciences, Incorporated, we have assembled these decisions into a time-phased flow, Figure III-94, which ties each decision into the other decisions which influence or are influenced by it. Polysciences has noted the various alternatives available at each decision point, and tabulated their current estimate of the probabilities that the given decision will be made according to each alternative. In addition, they have noted the particular alternative will be most satisfactory to them at this time.

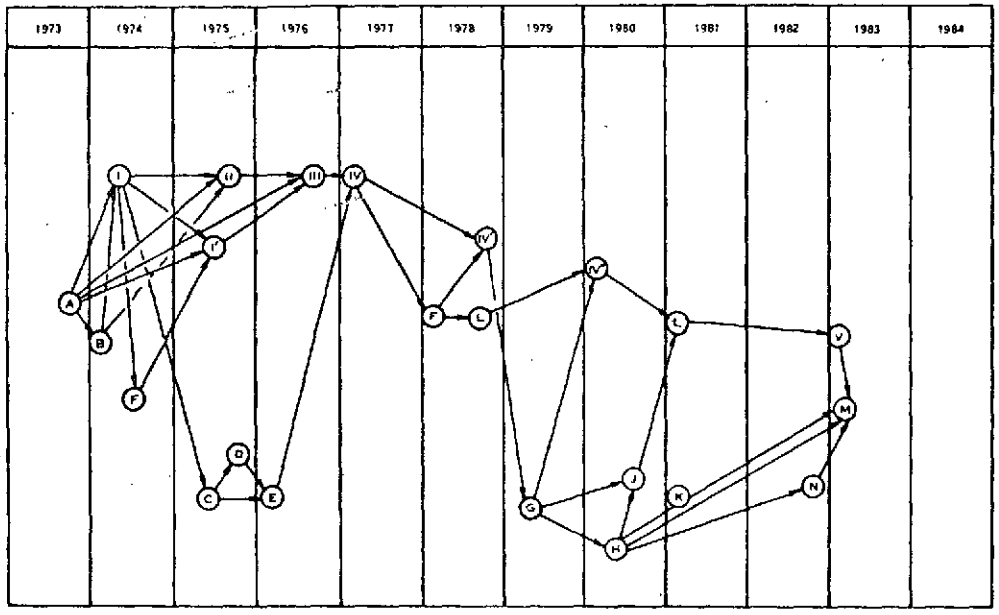
Polysciences is a small company, whose resources for "Blue Sky" research are limited. Decisions which require financial outlay for R&D, therefore, reflect a major dependence on NASA contracts for this effort. On that basis, Polysciences has indicated that patent rights and product ownership would likely reside with NASA, and Polyscience would expect to obtain licensing for production.

A key node in the decision flow is Item IV, which in 1977 should allow a decision as to the performance of the processing system equipment in ground tests. Acceptable performance of that equipment makes way for more advanced testing in short term Zero "G" facilities, and for requesting a flight opportunity for orbital testing.

Lesser nodes occur in decisions for funding phenomenological, concept, and environmental tests, early phenomenology test results, and in decisions on ownership and finances in 1980.

III.11.2 DECISION FLOW FOR PROCESSING TRANSPARENT OXIDES

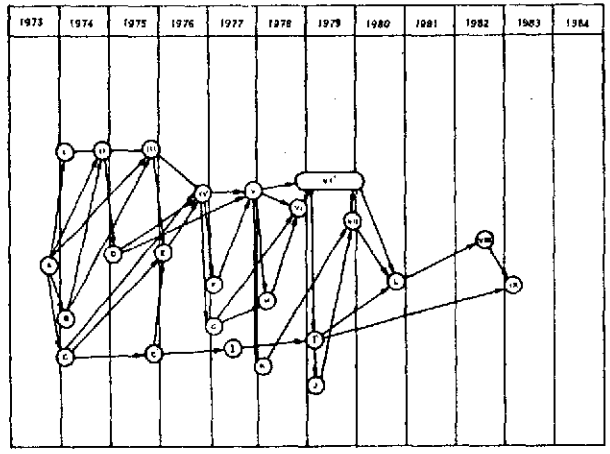
Corning Glass participated in establishing the decisions in Figure III-95. Question marks in the "probability" column, reflect the unavailability of sufficient data at this time to warrant an estimate.



KEY	DECISION TO BE MADE	ALTERNATES	PROBABILITY	BEST ALTERNATIVE
A.	FUNDING FOR EXP. SERIES I–III	A. IN-HOUSE FUNDING B. NASA FUNDING	10 90	•
B.	ASSIGNMENT OF PERSONNEL	A. REASSIGN AVAIL. PERSONNEL B. HIRE NEW PERSONNEL	30 70	•
C.	FUNDING FOR EXP. SERIES IV	A. NO FUNDING B. IN-HOUSE FUNDING C. NASA FUNDING	10 10 80	•
D.	NEW LAB SPACE	A. ADAPT EXISTING B. BUILD NEW, SEPARATE C. ADDITION TO PRESENT	20 10 70	•
E.	HIRING OF PERSONNEL	A. HIRE NEW PERSONNEL B. SUBCONTRACT	30 70	•

KEY	DECISION TO BE MADE	ALTERNATES	PROBABILITY	BEST ALTERNATIVE
F.	RESERVE FLIGHT OPPORTUNITY ON AUTOMATED SPACECRAFT	A. RESERVE B. CANNOT RESERVE	80 20	•
G.	PATENT RIGHTS FOR EQUIPMENT OF EXP. SERIES IV	A. POLYSC. OWN OUTRIGHT B. NASA OWNS C. POLYSC. LICENSE	30 30 40	•
H.	DEFINE OWNERSHIP/FINANCES OF COMMERCIAL SPACE PRODUCTION	A. POLYSC. OWNS B. NASA OWNS C. LICENSE TO CONTRACTORS	10 50 40	•
I.	FUNDING FOR DESIGN, FABRIC. AND TESTING OF EXP. SERIES V	A. IN-HOUSE FUNDING B. NASA FUNDING	10 90	•
K.	MAKE/BUY FOR INITIAL BIOLOGICALS	A. POLYSC. PRODUCE B. POLYSC. SUBCONTRACTS FOR PRODUCTION C. POLYSC. BUY FROM ESTAB. SOURCE	10 40 10	•
L.	RESERVE PL. ACCOMMODATION ON SHUTTLE	A. RESERVE B. CANNOT RESERVE	80 20	•
M.	INITIATE COMMERCIAL SPACE PRODUCTION	A. IN-HOUSE B. SUBCONTRACT FOR PROCESSING	50 50	•
I.	VALUE OF PHENOMENOLOGY RESULTS FROM TEST SERIES I	A. ALL SUPPORT SPACE PROCESSING B. SUFFICIENT SUPPORT C. INSUFFICIENT SUPPORT OF SPACE PROCESSING	20 70 10	•
II.	EVALUATION OF SEPARATION CONCEPTS IN TEST SERIES II	A. LIKELY CANDIDATE INDICATED B. NOT INDICATED	80 20	•
III.	EFFECTS OF NON-SPACE ENVIRONMENT IN TEST SERIES III	A. EFFECTS DETRIMENTAL B. NOT DETRIMENTAL	10 90	•
IV.	EVALUATION OF PERFORMANCE OF PROCESSING EQUIPMENT IN TEST SERIES IV	A. ACCEPTABLE B. UNACCEPTABLE	70 30	•
V.	PROOF OF PROCESS QUALITY, TIMING	A. SUFFICIENT FOR COMMERCIAL PROCESS B. INSUFFICIENT	80 20	•
I'	VERIFICATION OF PHENOMENOLOGY IN SPACECRAFT TEST	A. VERIFIED B. NOT VERIFIED	80 20	•
IV'	VERIFICATION OF SELECTED PROCESS EQUIPMENT DESIGN DATA IN ZERO "G" AIRCRAFT AND SHUTTLE FLIGHT	A. VERIFIED B. NOT VERIFIED	80 20	•
IV''	VERIFICATION OF PROCESS EQUIPMENT PERFORMANCE IN SHUTTLE FLIGHT	A. VERIFIED B. NOT VERIFIED	90 10	•

Figure III-94. Decision Flow For Separation Of Isoenzymes



KEY	DECISION TO BE MADE	ALTERNATIVES	PROBABILITY	BEST ALTERNATIVE
A.	FUNDING OF EXPERIMENTS 1, 2, 3	A. NASA FUNDING B. USER FUNDING	90 10	*
B.	ASSIGNMENT OF PERSONNEL	A. TRANSFER B. HIRE	10 90	*
C.	INITIAL DEFINITION OF LEGAL, FINANCIAL ARRANGEMENTS	A. PATENTS NASA OWNED B. PATENTS USER OWNED	20 80	*
C'	LEGAL, FINANCIAL ARRANGEMENTS SETTLED	A. MUTUALLY SATISFACTORY - CONTINUE B. NOT MUTUALLY SATISFACTORY-STOP	90 10	*
D.	PAYLOAD SPACE RESERVED ON AIRCRAFT, SOUNDING ROCKET	A. RESERVED B. NOT RESERVED	80 20	*
E.	FUNDING OF EXPERIMENT 4	A. NASA FUNDING B. USER FUNDING	50 50	*
F.	FUNDING OF EXPERIMENT 5	A. NASA FUNDING B. USER FUNDING	50 50	*
G.	PAYLOAD SPACE RESERVED FOR AUTOMATED SPACECRAFT TESTS	A. SPACE RESERVED B. SPACE NOT RESERVED	80 20	*

KEY	DECISION TO BE MADE	ALTERNATIVES	PROBABILITY	BEST ALTERNATIVE
H.	FUNDING OF EXPERIMENT 6	A. NASA FUNDING B. USER FUNDING	50 50	*
I.	PRELIMINARY EVALUATION OF POTENTIAL MARKET	A. FAVORABLE B. UNFAVORABLE	80 20	*
I.'	MARKET FORECAST	A. SUFFICIENTLY PROFITABLE B. NOT PROFITABLE	[illegible]	*
J.	FUNDING OF EXPERIMENT 7	A. NASA FUNDING B. USER FUNDING	20 80	*
K.	PAYLOAD SPACE RESERVED FOR SHUTTLE TESTS	A. RESERVED B. NOT RESERVED	90 10	*
L.	PROTOTYPE PLANT GO-AHEAD	A. NASA USER ARRANGEMENTS SETTLED B. NASA USER ARRANGEMENTS NOT ESTABLISHED	90 10	*
I.	CANDIDATE OXIDES SUFFICIENTLY CHARACTERIZED	A. SUFFICIENT B. INSUFFICIENT	95 05	*
II.	PROCESSING CONDITIONS VERIFIED IN LAB TESTS	A. VERIFIED B. MAINLY VERIFIED C. MAINLY NOT VERIFIED	10 80 10	*
III.	HEATING, MELTING, INITIAL POSITIONING METHODS VERIFIED IN DROP TOWER	A. VERIFIED B. NOT VERIFIED	75 25	*
IV.	HEATING, MELTING, POSITIONING METHODS VERIFIED IN ZERO 'G AIRCRAFT	A. VERIFIED B. NOT VERIFIED	75 25	*
V.	INITIAL FEASIBILITY OF PROCESS STEPS VERIFIED IN SOUNDING ROCKET TEST	A. COMPLETELY VERIFIED B. KEY STEPS VERIFIED C. KEY STEPS NOT VERIFIED	10 50 40	*
VI.	FEASIBILITY OF PROCESS STEPS VERIFIED IN AUTOMATED SPACE-CRAFT TESTS	A. COMPLETELY VERIFIED B. MAINLY VERIFIED C. MAINLY NOT VERIFIED	20 70 10	*
VI	EVALUATION OF MATERIALS PRODUCED IN AUTOMATED SPACE CRAFT, SOUNDING ROCKET, AIR-CRAFT TESTS, SHUTTLE	A. AMORPHOUS B. SATISFACTORY CRYSTALLINE C. UNSATISFACTORY CRYSTALLINE D. SELECT OTHER MATERIALS AND RECYCLE PROGRAM	[illegible]	*
VII	FEASIBILITY OF SPACE PROCESS VERIFIED IN SHUTTLE TESTS	A. VERIFIED B. NOT VERIFIED	90 10	*
VIII	PROTOTYPE PLANT OPERATION SATISFACTORY	A. OPERATION PER SPEC OR EASILY MODIFIED TO SO OPERATE B. NOT TO SPEC AND CANNOT BE SO MODIFIED	90 10	*
IX.	INITIATE PRODUCTION	A. YES B. NO	90 10	*

Figure III-95. Decision Flow For Transparent Oxides Processing

A key node appears to be the approximately one year of evaluation of transparent oxide specimens produced in short term and long term Zero "G" tests in 1979, 1980. While Corning would be satisfied with either amorphous versions or new crystalline structures, unsatisfactory results would imperil further effort.

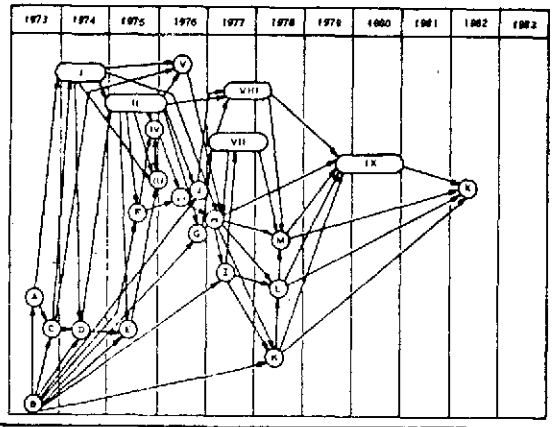
While Corning, too, looks to NASA for funding of early exploratory experiments, they presently consider later experiments and tests as having an equal probability of User or NASA funding.

III.11.3 DECISION FLOW FOR PROCESSING TUNGSTEN X-RAY TARGETS

GE's Medical Systems Business Division has structured the decision flow in Figure III-96, and the 1973 decisions reflect the on-going and imminent activities involved in phenomenological and concept testing. The User has already committed nearly $30,000 of his own funding for operational testing of promising products resulting from initial concept tests, and has accepted, for the time being, present NASA arrangements for proprietary rights and rights-in-data. More definitive decisions on such legal and financial arrangements are scheduled for 1978. For the relatively short period from 1973 to 1976 the present decision leads the User to give some consideration to NASA/User shared funding for experiments and tests, although favoring NASA funding.

Short term and long term Zero "G" tests in 1977, 1978 appear to hold key results for decisions to implement development of a production system.

Present unknowns in the process technology lead to unpredictability in 1977 decisions on feasibility and business prospects, while question marks on legal/financial arrangements reflect the above discussed need for further consideration.



KEY	DECISIONS TO BE MADE	ALTERNATIVES	PROBABILITIES	BEST ALTERNATIVE
A.	COMMITMENT OF LIMITED USER FUNDS TO SUPPORT NASA FUNDED LAB TESTING	A. COMMITMENT B. NO COMMITMENT	100 0	•
B.	SUFFICIENTLY INITIALLY DEFINED LEGAL FINANCIAL ISSUES	A. SUFFICIENT B. INSUFFICIENT	100 0	•
C.	FUNDING OF LAB TEST SERIES 1, 2, 3, 4, 5	A. NASA FUNDING B. USER FUNDING C. SHARED FUNDING	70 10 20	•
D.	FUNDING OF DROP TOWER TEST SERIES 6, 7	A. NASA FUNDING B. USER FUNDING C. SHARED FUNDING	80 ~ 0 20	•
E.	FUNDING APPROVAL OF ZERO G AIRCRAFT TEST SERIES 8	A. NASA FUNDING B. USER FUNDING, NASA APPROVAL C. SHARED FUNDING	70 10 20	•
F.	FUNDING APPROVAL OF SOUNDING ROCKET TEST SERIES 6	A. NASA FUNDING B. USER FUNDING, NASA APPROVAL C. SHARED FUNDING	70 ~ 0 30	•
G.	FUNDING, APPROVAL OF SOUNDING ROCKET TEST SERIES 7, 8	A. NASA FUNDING B. USER FUNDING, NASA APPROVAL C. SHARED FUNDING	70 10 20	•

KEY	DECISIONS TO BE MADE	ALTERNATIVES	PROBABILITIES	BEST ALTERNATIVE
H.	TECHNICAL FEASIBILITY OF PROJECT	A. FEASIBLE B. NOT FEASIBLE	? ?	•
J.	BUSINESS PROSPECTS	A. FAVORABLE B. UNFAVORABLE C. INDETERMINATE	? ? ?	•
J.	FUNDING, APPROVAL OF SPACECRAFT TEST SERIES 7, 8	A. NASA FUNDING B. USER FUNDING, NASA APPROVAL C. SHARED FUNDING	70 10 20	•
K.	SETTLEMENT OF USER/NASA LEGAL/FINANCIAL ARRANGEMENTS	A. MUTUALLY ACCEPTABLE B. NOT ACCEPTABLE	? ?	•
L.	CORPORATE FUNDING, FINANCING ARRANGEMENTS	A. CORPORATE APPROVAL B. DISAPPROVAL	? ?	•
M.	INITIATE PRODUCTION SYSTEM DEVELOPMENT	A. INITIATE B HOLD	90 10	•
I.	PHENOMENOLOGY TEST SERIES 1-5 SUFFICIENTLY CATEGORIZED IN LAB TESTS TO WARRANT CONTINUANCE	A. SUFFICIENT B. INSUFFICIENT	90 10	•
II.	TEST SERIES 6, 7 POSITIONING DEGASSING CONCEPTS INITIALLY TESTED IN DROP TOWER	A. FEASIBILITY SHOWN B. NOT SHOWN, POSITIVE RESULTS C NOT FEASIBLE, NEGATIVE RESULTS	50 30 20	•
III.	TEST SERIES 6, POSITIONING CONCEPTS TESTED IN ZERO G AIRCRAFT	A. FEASIBILITY SHOWN B. NOT SHOWN, POSITIVE RESULTS C. NOT SHOWN, NEGATIVE RESULTS	60 30 10	•
IV.	TEST SERIES 8, POSITIONING CONCEPT VERIFIED IN SOUNDING ROCKET	A VERIFIED B. NOT VERIFIED, CAN FIX C. NOT VERIFIED, NO FIX	70 20 10	•
V.	LAB TEST SERIES 4, 5 FINAL VERIFICATION OF DEGASSING, HEATING, MELTING PARAMETERS	A. VERIFIED B NOT VERIFIED	90 10	•
VI.	TEST SERIES 8, FINAL VERIFICATION OF POSITIONING CONCEPT IN SOUNDING ROCKET	A. VERIFIED B. NOT VERIFIED	90 10	•
VII.	SOUNDING ROCKET TEST SERIES 7, 8 VERIFICATION OF DEGASSING, MELTING, SUPERCOOLING CONCEPTS	A. VERIFIED B. NOT VERIFIED, CAN FIX C. NOT VERIFIED, NO FIX	70 20 10	•
VIII.	AUTOMATED SPACECRAFT TEST SERIES 7, 8 FINAL VERIFICATION OF DEGASSING, MELTING, SUPERCOOLING CONCEPTS	A. VERIFIED B. NOT VERIFIED, CAN FIX C. NOT VERIFIED, NO FIX	80 20 ~ 0	•
IX.	SPACE SHUTTLE TEST SERIES 9 FINALIZING PROCESS PARAMETERS	A. FINALIZED B. NOT FINALIZED, CAN FIX C. NOT FINALIZED, NO FIX	90 10 ~ 0	•
X.	SPACE SHUTTLE PROTOTYPE TEST	A. GO-AHEAD B. NO-GO	90 10	•

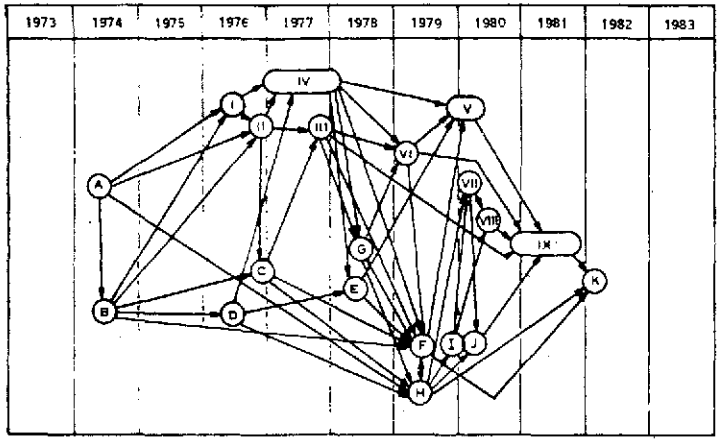
Figure III-96. Decision Flow For Tungsten X-Ray Target Processing

III.11.4 DECISION FLOW FOR FABRICATING SURFACE ACOUSTIC WAVE COMPONENTS

GE's Electronics Laboratory is already engaged in major efforts on surface acoustic wave components, although for lower frequencies. Their decision flow, Figure III-97, does not anticipate the need for technical decisions (on electron beam performance, and on crystal evaluation and growing) until 1976.

Their funding decisions reflect a dependence on NASA for experiment and test funds, thus, realistically, they show the high probability that resulting processing technology should be NASA (publicly) owned.

1977 decisions based on mask fabrication and x-ray litography test show major influence on decisions to approve subsequent tests and to establish a production organization.



KEY	DECISION TO BE MADE	ALTERNATIVES	PROBABILITIES	BEST ALTERNATIVE
A.	FUNDING OF LAB TESTS SERIES 1, 2, 3	A. NASA FUNDS B. USER FUNDS	90 10	*
B.	FORMATION OF USER DEVELOPMENT TEAM	A. GE B. OTHER	50 50	*
C.	GO-AHEAD, FUNDING OF SPACECRAFT TEST SERIES 6	A. OK, NASA FUNDS B. OK, USER FUNDS C. STOP PROGRAM	40 40 20	*
D.	FUNDING OF LAB TESTS SERIES 3, 7 AND ZERO "G" AIRCRAFT TEST SERIES 5	A. NASA FUNDS B. USER FUNDS	90 10	*
E.	APPROVAL OF SOUNDING ROCKET TEST SERIES 4, 3	A. APPROVED B. NOT APPROVED	90 10	*
F.	FORMATION OF PRODUCTION ORGANIZATION	A. GE B. OTHER C. NOT FORMED	50 50 ~ 0	*
G.	GO-AHEAD, FUNDING OF SPACECRAFT TEST SERIES 8	A. OK, NASA FUNDS B. OK, USER FUNDS C. STOP PROGRAM	40 40 20	*
H.	FINAL NASA, USER LEGAL, FINANCIAL ARRANGEMENTS	A. NASA FUNDING, PUBLIC OWNS B. USER FUNDING, USER OWNS	90 10	*
I.	SHUTTLE TEST SERIES 8 APPROVED, FUNDED	A. NASA FUNDING B. USER FUNDING C. NOT APPROVED	90 10 ~ 0	*

KEY	DECESION TO BE MADE	ALTERNATIVES	PROBABILITY	BEST ALTERNATIVE
J.	SHUTTLE PROTOTYPE TEST 8 APPROVED, FUNDED	A. NASA FUNDING B. USER FUNDING C. NOT APPROVED	90 10 ~ 0	*
K.	INITIATE FULL SCALE PRODUCTION	A. INITIATE B. HOLD	? ?	*
1.	SUCCESS OF LAB TESTS SERIES 1, 2	A. BOTH SATISFACTORY - CONTINUE B. 1ST SATISFACTORY, 2 NOT - CONTINUE C. 1ST NOT SATISFACTORY - END PROGRAM	? ? ?	*
II.	SUCCESS OF LAB TESTS SERIES 3	A. SATISFACTORY B. UNSATISFACTORY	80 20	*
III.	SUCCESS OF AUTOMATED SPACECRAFT TEST SERIES 3	A. SATISFACTORY B. UNSATISFACTORY	80 20	*
IV.	SUCCESS OF LAB AND ZERO "G" AIRCRAFT TESTS SERIES 5, 7	A. BOTH SATISFACTORY - CONTINUE B. 7 SATISFACTORY, 5 NOT - CONTINUE C. 7 NOT SATISFACTORY - END PROGRAM D. 7 NOT SATISFACTORY - ALTERNATE PRINT TECHNIQUE	70 10 ~ 0 20	*
V.	SOUNDING ROCKET TESTS VERIFY TESTS SERIES 4, 3	A. BOTH TECHNIQUES VERIFIED - CONTINUE B. 3 VERIFIED, 4 NOT -CONTINUE C. 3 NOT VERIFIED - END PROGRAM D. 3 NOT VERIFIED - ALTERNATE TECHNIQUE	70 20 ~ 0 10	*
VI.	SPACECRAFT TEST VERIFIES CONCEPT TEST SERIES 8	A. CONCEPTS VERIFIED FULLY B. CONCEPTS VERIFIED PARTIALLY, PROJECTED OPERATIONS OK C. CONCEPTS NOT VERIFIED	50 50 ~ 0	*
VII.	LAB TEST SERIES 6 VERIFIES MASK FABRICATION METHOD	A. VERIFIED B. PARTIALLY VERIFIED, FEASIBLE C. NOT VERIFIED	70 20 10	*
VIII.	SHUTTLE TEST SERIES 6 VERIFIES MASK FAB METHOD	A. VERIFIED B. PARTIALLY VERIFIED, FEASIBLE C. NOT VERIFIED	80 20 ~ 0	*
IX.	SHUTTLE TEST SERIES 8, PROTOTYPE PROOF	A. PROTOTYPE PROVED B. PROTOTYPE PARTIALLY PROVED, FIX POSSIBLE C. PROTOTYPE DISPROVED	50 50 ~ 0	*

Figure III-97. Decision Flow For Fabrication Of Surface Acoustic Wave Components

SECTION IV

CONCLUSIONS AND RECOMMENDATIONS

Completion of the Phase II study has provided us with an education in the methods, hopes and fears of a portion of the non-aerospace community, and a data bank of information on key aspects of developing the capabilities to space process four commercially-oriented products. From that education and data, we are able to draw the conclusions, and offer the recommendations discussed below.

IV.1 CONCLUSIONS

We believe the study has been successful. In its non-technical aspects, the study has maintained the previously developed rapport between the segments of the non-aerospace community and the aerospace community represented by participants in the study and by NASA. Furthermore, during the study we have had the opportunity to utilize information gathered in this study to "spread the gospel" on space processing to our non-aerospace corporate officers, the Congressional House Committee on Science and Astronautics, and, via television, to the public.

Technically, the study has met the rationale laid down for us by NASA a year ago:

- Emphasis on Specific Ideas - We not only maintained an undeviating baseline in the four Ideas selected for the study, but retained the same type of specificity in documenting results of each task.
- Definition of Specific Requirements - Where sufficient data existed, we documented experiment and test requirements in detail, listing temperatures, pressures, times, functions, etc. Where such data was not available we obtained experts' judgement, when possible. Few requirements we have noted do not meet the criterion of specificity.

As a result of the above, we have documented a number of key data points:

1. 26 major alternatives, and over 100 lesser alternatives in approaches to processing the four products.

   Our conclusions, after selecting an approach for processing each product are:

- Little hard data presently exists for assessing the alternatives.

- Projection of 1980 technology is a "shaky" business.

- Judgement and "feel" for such projections, without experimental data, vary from expert to expert.

    o Although the required selection of an approach for processing each product was performed, we must note that the selections were speculative.

2. The requirements for 66 series of experiments and tests were documented. Nearly 30 percent of these call for the "low cost testing" facilities discussed in Section III.9.5.

   Our conclusion here is that:

   - Significant experimental work toward commercial space processing could be initiated soon at relatively low cost.

3. The requirements for 11 series of experiments and tests on Shuttle/Spacelab comprise more than 200 test runs.

   Our conclusion, based on the level of testing required for the four products under study is:

   - By 1980, the full complement of products under development via space processing will require considerably more than the 12 space processing Spacelabs shown in the NASA Mission and Payload models.

4. Timelines and milestones for development of the space processing systems of the four products all fit the 1973 to 1982 timeframe "comfortably".

   Our conclusion is that:

   - The schedules are sufficiently flexible to account for experimental checking of key alternatives in all approaches, rescheduling of orbital tests to Shuttle operational dates, and accommodation of moderate redirection in the event of negative results in early experiments and tests.

5. Decision flows possess, for most of the products under study, major "nodes", wherein critical decisions will exert profound influence on subsequent development. Most such nodes are technical in nature, but User evaluation of decision alternatives, probabilities and desires reflect major concern over NASA/User legal/financial arrangements.

   Our conclusion is that:

   - This concern, first noted during the Phase I study, remains unresolved, and will eventually require open, equitable arrangements.

IV.2 RECOMMENDATIONS

The status of space processing applications has been formally and informally reviewed with the NASA Steering Group for this study, members of the NASA's Materials Advisory Board, members of the NASA's Office of Applications, members of the NASA-MSFC Preliminary Design and Process Engineering Staffs, as well as the Users participating in this study in order to determine whether further effort related to this study is warranted, and what the thrust of that effort should be.

A number of key points have been brought out in these reviews:

1. The Phase I effort, which touched very much less than 1% of the potential non-aerospace User community, developed a two-way flow of information with 80 organizations. The approximately 12 organizations whose Ideas for development or production in space appeared to possess a high degree of merit were motivated to further participation, while the rest have had essentially no further contacts.

2. Of the organizations whose 12 high merit Ideas were identified in Phase I, only 4 were exposed to further contacts in Phase II, leaving 8 rather highly motivated organizations with no further formal contacts.

3. During the course of the Phase II study, several User-promoted contacts were made with organizations not previously involved in the study. These organizations had had their attention directed to the aims and results of the study by various individuals, and were sufficiently interested to initiate telephone conversations, set up meetings, and visit our facility. Several Ideas of potential value resulted; e.g., Tungsten Carbide Valves, Beryllium Composites, etc.

4. Even those four organizations who participated in Phases I and II have been involved in only very preliminary technical and planning aspects of commercial space processing. They, the Space Division study, and NASA, all recognize that additional depth, both technical and planning, would be useful in preparing future plans - both NASA's and User's.

Based on these points, we offer the following recommendations for further activity in the area of Beneficial Uses of Space:

- Perform a Business Planning Study. Based on item 4. above, it would appear to be worthwhile, from the viewpoint of further encouraging commercial utilization of space processing, and from the viewpoint of

furthering mutual NASA/User understanding of each others' modes of operation, constraints and impelling concerns, to carry out a Business Planning Study. Such a study should aim at assessing the validity of available standard business planning and market forecasting methods for determining the cost and resource requirements of space processing the four specific commercial products studied during Phase II.

As a first step, the Business Planning Study should utilize the four User organizations involved in Phase II to prepare business plans and market forecasts for these products.

With such information and the Phase II results, combined with the results of recent and concurrent studies on space processing equipment, the next step should identify Shuttle payload equipment, Sortie flights, Shuttle/Spacelab resources, and space program costs to accomplish the R&D phases of the business plans.

Finally, estimates of required flight system resources should be provided for the pilot production and full-scale manufacturing of the four product lines.

Figure IV-1 depicts the logic of such a planning study, proposed as a Phase III effort in this study.

- Foster identified interests in space processing among non-aerospace Users. Based on Items 1. and 3. above, efforts to maintain the interest of the body of potential Users identified in Phase I of this study and to uncover such potential Users should be undertaken with low-keyed, positive steps.

Typically, these organizations would be interested in Space Processing results derived from Skylab, NASA Centers, such as the Astronautics Laboratory and the Process Engineering Laboratory at Marshall Space Flight Center, NASA-contracted work at National Buerau of Standards and at Aerospace companies. Such interests should be maintained by a steady flow of information on both actual processing results and on results of studies such as this. Technically oriented news letters

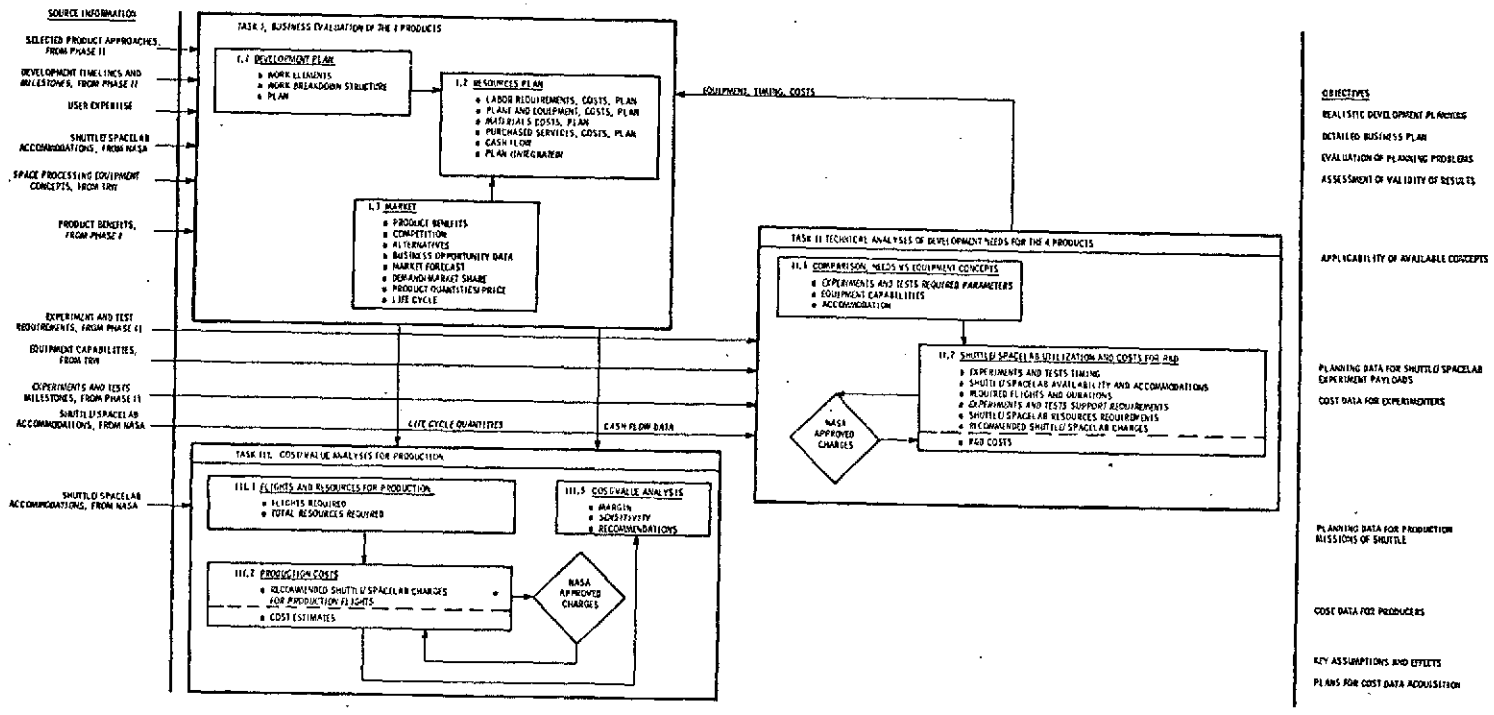


Figure IV-1. Business Planning Study

and/or specific news releases stressing key experimental and study results, and emphasizing potential new User-oriented products, requirements for new techniques or materials, or subjects for up-coming investigations, would serve the required purpose. In time, such an interest could grow to a two-way flow of information as these potential Users found products or processes of interest, potential solutions to problems, etc.

In a wider range, such an activity, preceded by a User survey such as that carried out in Phase I, could elicit response from an increased spectrum of the potential User community. Thus we recommend that NASA:

- Initiate an extension of the Phase I study to identify additional Beneficial Uses of Space. Items 1. and 3. indicate that there are many more potential space processing Users in the vast non-aerospace community. With the techniques developed, and the lessons learned in the Phase I study, the identification of additional potential Users will be a much more efficient process.

- Initiate experiment programs. Sections III.5, III.6, III.7 and III.8 document a number of relatively straightforward ground laboratory experiments and tests that could be initiated now. These experiments, aimed at authentic products rather than at a search for knowledge, would be solid evidence that space processing is "Down To Earth".

In addition, NASA must find a way to attract additional members of the non-aerospace community to participate in opportunities for space processing research - in unique ground laboratories, low cost space-simulating facilities such as Drop Towers, Zero "G" Aircraft Flights, Sounding Rockets, and eventually the Space Shuttle. We have reviewed current procedures and practices for establishing such opportunities, and find that an evaluation of these methods is needed. Announcement of opportunities, and documentation need to be analyzed from the viewpoint of impact on the non-aerospace User. Modifications should be considered, which provide for wider dissemination of announcements, simplification of paperwork, and interfacing support which adapt space program methods and equipment to non-aerospace methods and equipment.

A major effort should be undertaken to develop a "Low Cost Testing" plan. The existence of a number of facilities in which some aspect of space processing can be tested has been documented in Section III.9.5. The low cost (compared to past and present space facilities) for testing in such facilities, and their availability during the hiatus between Skylab and Shuttle poses them as attractive means of carrying on an active space processing program in the next few years. What is needed is an activity which searches out potential Users of these facilities, documents and integrates their requirements, educates the Users (especially non-aerospace Users) to the capabilities and limitations of the facilities, and combines the resulting data into a comprehensive plan.

Implementation of such a plan would provide an excellent baseline for an integrated space processing program.